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A PROTOTYPE VISCOMETER FOR TIME-DEPENDENT FLUIDS

by

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A THESIS

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ABSTRACT

A search of the current literature has revealed a decided lack of suitable instruments for measuring time-dependent non-Newtonian fluid properties. The basic principles utilized in measuring fluid properties are discussed and the design and operating characteristics of a number of the various rheometers and viscometers available are reviewed.

The application of rheological data to pipeline design is discussed, and a new instrument, designed specifically for measuring thixotropic behavior, is proposed. The new instrument, which consists essentially of four cup and bob assemblies rotating at four different speeds, has a novel cup assembly design which permits a fresh sample to enter the annular space between the cup and bob whenever the instrument is charged with a fluid to be tested. The data from the instrument will be obtained in the form of shear stress decay curves, which in turn may be utilized for pipeline design calculations.

The design details and calibration of a prototype viscometer, built to test the features of the new instrument, are presented. During the calibration of the prototype viscometer, a number of design weaknesses were observed.

These weaknesses are discussed along with the suggested design improvements to be incorporated in an industrial model.

With the prototype viscometer, it has been shown that the basic principles of the viscometer are satisfactory. The program, therefore, will be continued with the construction of the proposed viscometer, suitable for field testing, and prediction of pipeline pressure drops for comparison with observed pressure drops.

ACKNOWLEDGMENTS

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Special thanks are also due to Mr. F. E. Butz and the staff of the Chemical Engineering Shop for the speed with which they completed the prototype viscometer and for their many helpful suggestions during the construction of the equipment; to Mr. D. R. Sutherland for determining the viscosities of the test fluids, and to my wife for typing the manuscript.

TABLE OF CONTENTS

	Page
TABLE OF CONTENTS	i
LIST OF TABLES	iii
LIST OF FIGURES	iv
I INTRODUCTION	1
A Rheological Types of Fluid Behavior	1
B Historical Developments in Rheology	7
C Engineering Application of Rheological Data	10
II VISCOMETER TYPES	13
A General	13
B Capillary Viscometers	14
1. Glass Capillary Viscometers	16
2. Orifice Viscometers	25
3. Cylinder-Piston Viscometers	26
C Falling-Body Viscometers	31
1. Falling-Ball Viscometers	33
2. Rolling-Ball Viscometers	34
3. Rising-Bubble Viscometers	36
4. Falling-Piston Viscometers	36
5. Float Viscometers	39
D Rotational Viscometers	40
1. Concentric Cylinder System	41
2. Rotating Cup Viscometers	46
3. Rotating Bob Viscometers	50
4. Cone and Plate Viscometers	57
E Vibrational Viscometers	60
F Parallel-Plate Viscometers	62
G Sliding-Plate Viscometers	62
III DETERMINATION OF THIXOTROPIC BEHAVIOR	66
IV APPLICATION OF RHEOLOGICAL DATA TO PIPELINE DESIGN	68
V PROPOSED VISCOMETER FOR TIME-DEPENDENT FLUIDS	73
VI PROTOTYPE VISCOMETER DESIGN	80
VII PROTOTYPE VISCOMETER CALIBRATION	98
VIII DISCUSSION OF OPERATION AND RECOMMENDATIONS	108
IX FUTURE WORK	115

X CONCLUSIONS 116

XI NOMENCLATURE 118

XII BIBLIOGRAPHY 119

APPENDICES

I Detail Drawings

II Sample Calculations

III Determination of Instrument Constant From Previous Work

LIST OF TABLES

TABLE		
1	Test Fluid Viscosities	98
2	Calibration Data	101
3	Calculation of Instrument Constant	102
4	Instrument Constant	103
5	Effect of Master Pulse Firing Early	111

LIST OF FIGURES

FIGURE		
1	Graphical Representation of Time-Dependent Behavior	6
2	Thorpe and Rodger's Viscometer	18
3	Ostwald Viscometer	19
4	Common Glass Capillary Viscometers	22
5	Cragg and Van Oene Viscometer	24
6	Saybolt Viscometer	27
7	MCER Capillary Viscometer Extruder Assembly	29
8	Westover High Pressure Rheometer	30
9	Hoeppler Rolling-Ball Viscometer	35
10	Norcross Viscometer	37
11	Hatschek's Concentric Cylinder Apparatus	48
12	Searle's Concentric Cylinder Apparatus	51
13	Haake Rotovisco Viscometer Measuring Head	53
14	Merrill-Brookfield Viscometer Measuring Head	56
15	Ferranti-Shirley Plate and Cone Viscometer	59
16	Band Viscometer	64
17	Prototype Viscometer	81
18	Prototype Viscometer- Lower Section	82
19	Prototype Viscometer - Cup & Bob	83
20	Prototype Viscometer - Plan	84
21	Prototype Viscometer - Front Elevation	85
22	Prototype Viscometer - Section I-I	86
23	Prototype Viscometer - Section A-A	87
24	Prototype Viscometer - Section B-B	88
25	Prototype Viscometer - Section C-C	89
26	Prototype Viscometer - Section D-D	90
27	Electrical Circuits - Block Diagrams	95
28	Deflection Angle versus Cup Speed	105
29	Effect of Improper Zeroing of the Instrument	112

A PROTOTYPE VISCOMETER FOR TIME-DEPENDENT FLUIDS

INTRODUCTION

Rheological Types of Fluid Behavior

Although man has always been concerned with problems of fluid flow, the science of the deformation and flow of matter, known as Rheology, has been given systematic study only recently. The modern era of rheology was begun in 1687 when Newton in his Philosophiae Naturalis Principia Mathematica postulated his hypothesis concerning the magnitude of the force required to overcome viscous resistance. This hypothesis which states that the shear force per unit area is proportional to the negative of the local velocity gradient is the basis of the whole theory and practice of viscometry. Mathematically, Newton's hypothesis or Law of Viscosity may be written as:

$$\tau = \frac{-\mu}{g_c} \frac{dv}{dx} \qquad (1)$$

where τ = shear force per unit area or shear stress, lb_f/ft^2

$\frac{dv}{dx}$ = velocity gradient or shear rate, sec^{-1}

μ = constant of proportionality known as the viscosity, $lb_m/sec\ ft$

and g_c = dimensional conversion factor, $lb_m ft/lb_f sec^2$

Those fluids, which in the laminar-flow region exhibit a viscosity that depends only on temperature and pressure and is independent of the rate of shear, are classed as Newtonian. The flow curve relating the shear stress and rate of shear for these fluids is a straight line of slope $\frac{\mu}{g_c}$. The fluid, therefore, is completely characterized by a single physical property, the viscosity.

Non-Newtonian fluids (R-64) are those which, at a given temperature and pressure, do not obey the direct proportionality between shear stress and shear rate given by equation (1). These real fluids may be classified into three broad categories;

(1) Fluids with properties independent of time or duration of shear.

(2) Complex fluids for which the relationship between shear stress and shear rate depends upon the duration of shear.

(3) Systems which have characteristics of both solids and fluids and exhibit partial elastic recovery after deformation.

Time-independent non-Newtonian fluids are classified according to their shear stress-shear rate curve. The types which may be encountered are: the Bingham Plastic, the Pseudoplastic, and the Dilatant fluids.

Bingham plastics exhibit a yield stress below which

elastic deformation takes place rather than flow, and above which the shear stress, in excess of the yield value, is directly proportional to the shear rate. The Bingham plastic is defined mathematically by:

$$\tau - \tau_y = \frac{-\eta}{g_c} \frac{dv}{dx} \qquad (2)$$

where τ_y is the yield stress lb_f/ft^2

and η is the coefficient of rigidity.

Pseudoplastic fluids show no yield value. The typical flow curve for these materials indicates that the ratio of the shear stress to the rate of shear, which may be termed the apparent viscosity, decreases systematically as the shear rate is increased.

Dilatant fluids are similar to pseudoplastics in that they show no yield stress, however, the apparent viscosity of these materials increases with increasing rates of shear.

The flow curves for Pseudoplastic and for Dilatant fluids are often approximated by a power law relationship between the shear stress and the shear rate:

$$\tau = \frac{-K}{g_c} \left(\frac{dv}{dx}\right)^n \qquad (3)$$

The flow behavior index, n, characterizes the extent of the deviation of the fluid from Newtonian behavior. The consistency index, K, characterizes the consistency or "thickness" of the fluid. It is analogous to the viscosity of a Newtonian fluid.

With some more complex fluids it is found that the apparent viscosity depends not only on the rate of shear, but also on the prior thermal and/or shear treatment of the fluid. These time-dependent fluids are classed as thixotropic if the shear stress decreases with the duration of shear at a constant rate of shear, and are classed as rheopectic where the reverse occurs. The relationship between the shear stress and the shear rate for a typical thixotropic fluid is shown in Figure 1a on arithmetic coordinates. The relationship for a rheopectic fluid is shown in Figure 1b.

If a thixotropic material is sheared at a constant rate after a period of rest, the structure of the material will be progressively broken down and the apparent viscosity will decrease with time to some lower limiting value. This type of behavior leads to a kind of hysteresis loop on a plot of shear stress versus instantaneous shear rate if the shear rate is varied continuously, at a predetermined rate, from zero to a maximum and back to zero again. Typical hysteresis loops are shown in Figure 1c.

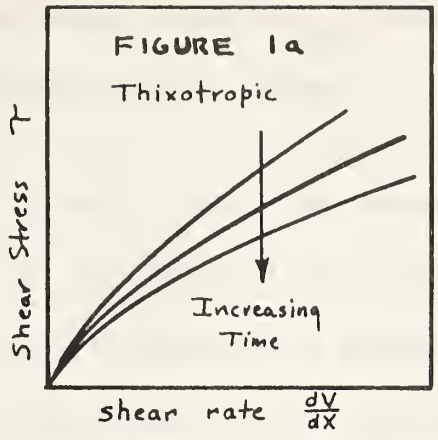
The presence of thixotropy may conveniently be determined with the use of a concentric cylinder viscometer (to be discussed later). If the fluid confined in the instrument has been resting for a long time, and then subjected to a constant shear rate, the shear stress would decrease with time as shown

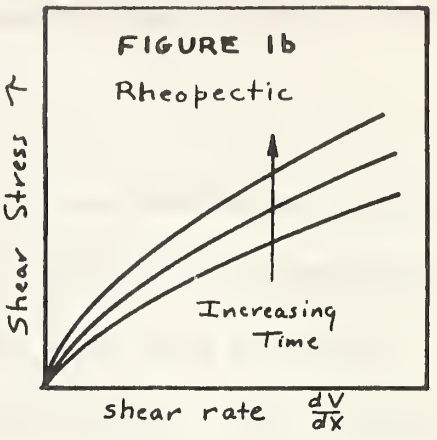
in Figure 1d. The rate of decrease and the final shear stress would both depend on the rate of shear and hence a family of curves would be obtained for various shear rates. Curves of this nature will completely describe a thixotropic fluid and may be applied to equipment design (R-23).

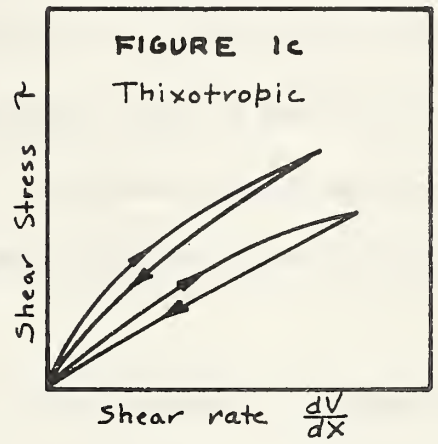
Time-dependency may also be indicated by a capillary viscometer (R-37). More thixotropic breakdown would be indicated with longer pipes (longer times of shear) and smaller diameters (higher rates of shear). Therefore, if the shear stress is plotted against the apparent rate of shear, for various capillary lengths and diameters, a plot similar to Figure 1e would be obtained. Rheopectic fluids show exactly the opposite effect as shown in Figure 1f. In both Figures 1e and 1f, the shear stress is defined as $D\Delta P/4L$ and the apparent shear rate as $8V/D$ where D is the capillary diameter, L is the capillary length, ΔP is the pressure drop across the capillary, and V is the average velocity of the fluid.

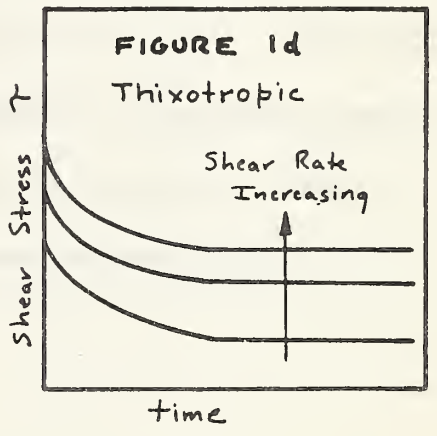
Many time-dependent fluids often exhibit an initial yield value somewhat like the yield value of a Bingham plastic. Unlike a Bingham plastic, however, the initial yield value of a thixotropic fluid is destroyed as soon as the fluid is sheared and is only regained after allowing the structure to reform by resting the fluid for a long time at zero shear rate.

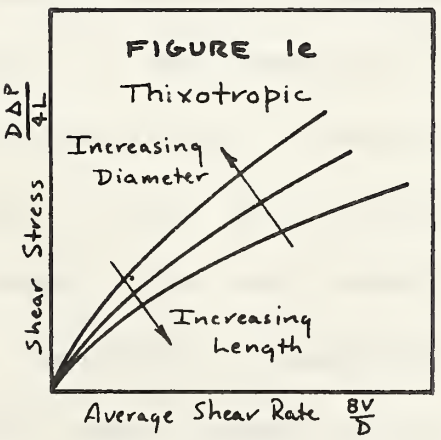
FIGURE 1 GRAPHICAL REPRESENTATION OF TIME-DEPENDENT BEHAVIOR

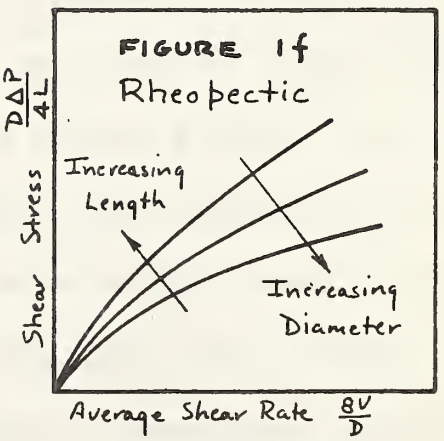












Some fluids, however, exhibit a reduced yet finite yield value even after shearing. These time-dependent fluids are termed "false-bodies" (R-64).

The fluids in the last category of non-Newtonian fluids are known as the viscoelastic fluids. These fluids because of their elasticity exhibit a tendency to flow in a direction normal to the direction of the shearing stress. This tendency is the well known "Weissenberg effect" (R-62) and has been used (R-47) to characterize viscoelastic liquids. The study of viscoelasticity (R-4,8 and 17) has become increasingly more important in recent years because of the interest shown in high polymer systems, most of which are viscoelastic.

Historical Developments in Rheology

The early history of rheology is obscure. As early as about 3000 B. C. the Sumerians used, as a unit of weight, the quantity of water which flowed from a standard vessel in a given time. In 1540 B. C., an Egyptian named Amenemhet invented a water clock that consisted of a conical vessel with a hole in the bottom. Amenemhet's work, reported by Scott-Blair (R-50) even allowed for the drop in temperature and consequent rise in viscosity which must have taken place during the night.

Scott-Blair (R-50) reports that there is little in the way of rheological developments recorded in Greek and Roman writings, although the Indians had developed rheological science very thoroughly by about the first century A. D. During the Dark Ages there were no important developments in Europe until the coming of Leonardo da Vinci (1452-1519). He studied (R-13) the efflux of water through orifices and its flow in channels. In 1638, Galileo published his Discourses Concerning Two New Sciences, in which he discussed the cohesion of ropes, the causes of fractures in solids, and the shearing of beams. Later, in 1676, Hooke proposed his law which states that, for an ideal elastic body, the deformation under stress is directly proportional to the deforming force.

Following Newton's fundamental work in 1687, over one hundred years elapsed before Navier (R-39) stated the general equations for motion in real fluids. Similar equations were deduced by Poisson (R-44) and Stokes (R-54). From an experimental point-of-view, Hagen (R-27) attempted to determine the law of flow through cylindrical tubes. His work was completely overshadowed by the classical researches of Poiseuille (R-43) in 1846. Poiseuille, using water flowing through very fine glass tubes, discovered the relation between flow rate and pressure drop for capillary flow.

Following Poiseuille, Wiedemann (R-61) and later Hagenback (R-28) deduced a theoretical formula for Poiseuille's discovery, based on Newton's hypothesis. Their formula known as the Hagen-Poiseuille Law can be written as follows:

$$\frac{\Delta P}{L} = \frac{8\mu Q}{g_c \pi a^4} = \frac{8\mu V}{g_c a^2} \qquad (4)$$

where $\frac{\Delta P}{L}$ = pressure drop per foot of capillary lb_f/ft^3

Q = flow rate ft^3/sec

V = average velocity ft/sec

a = radius of the capillary, ft

μ = viscosity, $lb_m/sec\ ft$

g_c = dimensional conversion factor $lbm\ ft/lb_f\ sec^2$

The work of Poiseuille was the foundation of capillary viscometry which became a reliable means of measuring the flow properties of fluids. Until 1890, capillary flow was the only widely used technique in viscometry. In that year, Couette (R-12) devised a new method of measuring viscosity based on a system of two concentric cylinders, one rotating and the other at rest.

Twenty-three years later, Hatcheck (R-27,30) described a modified version of the Couette viscometer designed to minimize the cylinder end effects with very low viscosity fluids.

Shortly thereafter the MacMichael (R-33), and the Stormer (R-56) viscometers were developed.

Since 1940, numerous viscometer designs have appeared in the literature. As a result there are many instruments available to the practical rheologist. In fact, one reference (R-5) lists 56 commercially available designs, and another (R-60) lists 75. There are also many more not commercially available. A few of the more common viscometers will be discussed in a later section.

Engineering Application of Rheological Data

From an engineering point-of-view, the relationships existing between shear stress, shear rate and previous thermal and shear history must be known in order to make accurate pipeline design calculations. For Newtonian fluids, the procedures for calculating the pressure drop-flow rate relationships as a function of line size and fluid properties are well established for both laminar and turbulent flow conditions. Much less is known for non-Newtonian fluids, although in the simpler cases methods for designing pipelines have been developed (R-22,37).

Normally the consistency of a non-Newtonian fluid must be defined with respect to shear rate, shear stress and at

times, previous thermal and shear history. Some attempts have been made to use a single apparent viscosity and to treat the fluid as though it were a Newtonian. This method is sometimes satisfactory, however, a difficulty arises in the choice of the apparent viscosity. Other methods of pipeline design for non-Newtonian fluids are discussed by Metzner (R-37).

Govier and Ritter (R-23) presented methods of applying rheological data of thixotropic crude oils to the design of crude oil pipelines. Their method, to be discussed later, requires the determination of the shear stress as a function of the duration of shear at a minimum of three shear rates.

A review of the many viscometers discussed in the literature has revealed that there are no instruments available to simultaneously determine the shear stress-duration of shear relationship at more than one shear rate, a feature that would be highly desirable when studying thixotropy. Many of the viscometers reviewed have some very desirable features and these are discussed with particular emphasis on the suitability of the instruments for determining the consistency of thixotropic fluids.

In this study, a new instrument, designed specifically for studying the flow behavior properties of time-dependent

fluids, is proposed. In particular a prototype viscometer has been constructed and tested in preparation for the construction of a proposed model suitable for field installation. The application of data obtainable from this new instrument should ensure greater accuracy in the design of pipelines used for transporting time-dependent fluids.

VISCOMETER TYPES

General

The selection of a suitable instrument for the determination of rheological properties depends on the nature of the fluid in question and the type of information desired. There have been many types of viscometers designed and used by individual rheologists. In fact, until recently nearly all investigators constructed their own viscometers. Today there appears to be little reason to construct special viscometers for studying time-independent flow properties since many commercial instruments are extremely good. However, better techniques and instruments are needed for investigating fluids which exhibit time-dependent flow properties.

Most early viscometers, many of which are still used in standard industrial tests, give single-point measurements. That is, rather than giving the relationship between shearing stress and rate of shear over a range of values, only a single point on the flow curve is obtained from the measurement. Such single-point measurements are perfectly adequate for measuring the flow behavior properties of Newtonian fluids, since by definition the viscosity of a Newtonian fluid is independent of the rate of shear.

A single-point instrument, however, is not satisfactory for studying non-Newtonian or complex fluids. With these fluids, the "viscosity" is not constant at a given temperature and pressure. Therefore, to adequately describe the flow behavior properties, the entire shear stress-shear rate curve must be determined.

Viscometers might be classified in several ways. One means of distinguishing between them (R-1) is to group them generally according to the measurements employed: (1) The rate of fluid flow in tubes, (2) the rate of motion of solids through subject media, (3) the torque required to rotate a solid in the media, (4) the distortion of solids by forces transmitted through the media from other solids in motion, (5) the damping effect of a medium on a solid vibrating in it, (6) the distortion of media under compression, and (7) the rate of displacement of a sliding plate.

Capillary Viscometers

A capillary viscometer consists of five essential parts: (1) a fluid storage chamber, (2) a capillary of known dimensions (3) a means for controlling and measuring the applied pressure, (4) a unit for controlling the temperature, and (5) a device for determining the flow rate. Devices of this sort, which include the glass capillary, the orifice and the cylinder-

piston viscometers, have been in general use longest and still are the most popular viscometers since they are relatively inexpensive and convenient. They are not readily adaptable to rheological studies.

With all instruments of this group, the viscosity of the test media is determined from the measured volumetric flow rate, the applied pressure, and the instrument dimensions by application of the Hagen-Poiseuille Law. With the glass capillary viscometers, the time required to transfer a known volume of sample from one part of the apparatus through a tube to another part is measured. With the orifice viscometer, the time required to discharge a measured amount of sample through a capillary exit is measured. With the cylinder-piston viscometer, the weight of sample extruded through a short capillary in a unit time is determined.

Since all calculations involving capillary viscometers are based on the Hagen-Poiseuille Law, and because of the assumptions made in deriving the Hagen-Poiseuille Law (R-7), the following conditions are assumed to apply to the fluid flow in a capillary viscometer:

(1) the flow must be steady

(2) the velocity must have no radial or tangential components

(3) the axial velocity is a function of the distance from the axis alone

(4) there is no slippage at the wall

(5) the tube is sufficiently long that end effects are negligible

(6) the fluid is incompressible

(7) isothermal conditions prevail throughout

(8) the viscosity does not change appreciably with the change in pressure down the tube.

In most cases all of the above conditions do not apply and some corrections will have to be made to represent the real case. For instance, in some cases the measured pressure drop must be corrected for pressure losses at the entrance or exit of the tube (ie. end effect), or for the pressure due to the head of liquid in the tube and material storage chamber. Many capillary viscometers have been proposed, some with unique features designed to meet special needs. A few of the many capillary viscometers are now discussed.

Glass Capillary Viscometers

With glass capillary viscometers the driving force is usually the hydrostatic head of the liquid sample itself. For this reason, kinematic viscosity (absolute viscosity divided by fluid density) is measured directly. Most of the

glass capillary viscometers are limited to low viscosity fluids although some instruments may be operated with the application of an external pressure, thus increasing the range of the viscometer.

The first convenient glass viscometer to be devised following the work of Poiseuille was designed by Thorpe and Rodger (R-57). This instrument, shown in Figure 2, is the prototype of many viscometers used for the precise determination of viscosity. The horizontal capillary tube is fused into the glass sleeves connecting the bulbs L and R. Each vertical limb has marks placed at m_1, m_2, and K_1 on the left, and at m_3, m_4 and K_2 on the right. The test fluid is charged to the instrument and the sample volume adjusted so that the upper level initially is at the top of the tube in bulb T_1 (or T_2) and the lower level at mark K_2 (or K_1). The liquid is then forced through the capillary by air pressure measured by a suitable manometer, and the time taken for the upper level of the fluid to pass from m_1 to m_2 (or m_3 to m_4) is measured. Readings are then taken in the opposite limb and the two sets averaged.

The standard practice with many present day glass capillary viscometers, which are based on a viscometer originally used by Ostwald (R-41), is to calculate the fluid viscosity

FIGURE 2 THORPE AND RODGER'S VISCOMETER

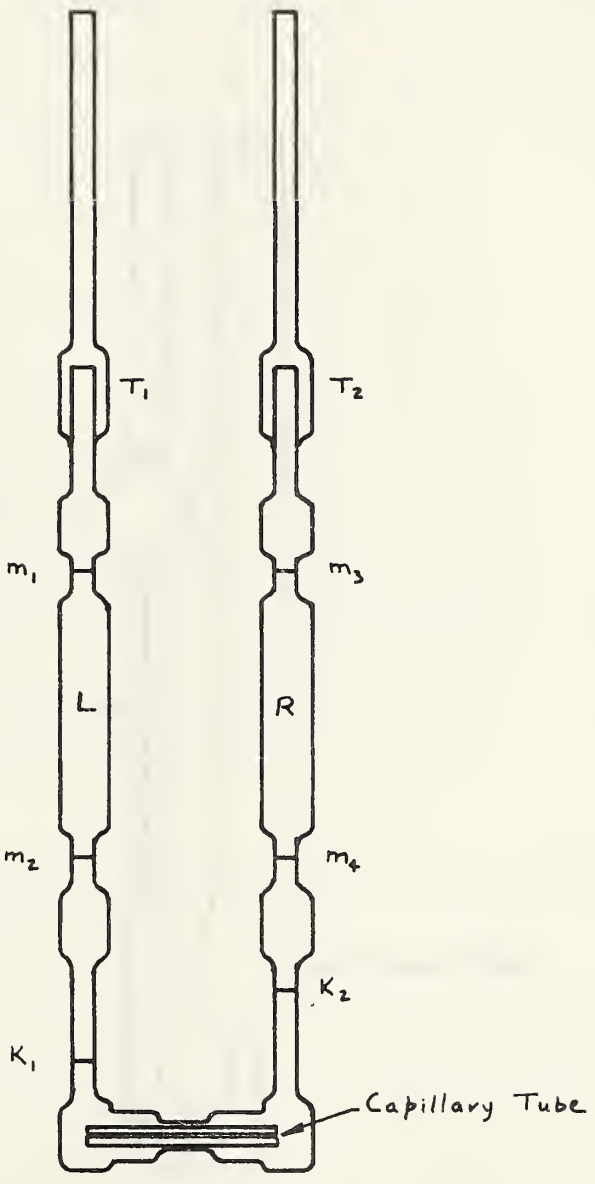
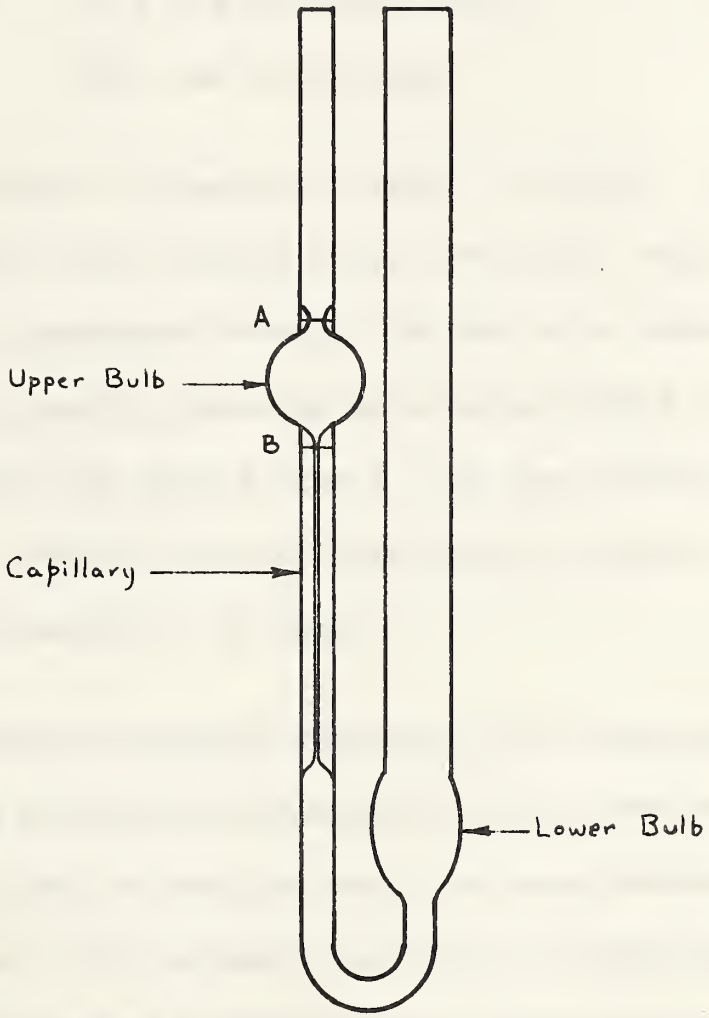


FIGURE 3 OSTWALD VISCOMETER



using the relationship:

$$\mu = k\rho t$$

where k = a constant obtained by calibrating the instrument with a liquid of known viscosity

ρ = the liquid density

t = the efflux time

The Ostwald viscometer as shown in Figure 3 is composed of a reservoir bulb located above a vertical capillary whose lower end is connected through a U-tube to a lower receiving bulb. It is usually operated by drawing liquid up into the reservoir bulb and beyond mark A, and then allowing the liquid to flow back through the capillary while recording the time for it to fall from mark A to mark B.

This original Ostwald viscometer has been modified in many ways to minimize certain undesirable effects in viscosity measurements and to meet the specific requirements of certain test liquids. For instance, in order to reduce the error in the mean head caused by deviation of the viscometer from the vertical, Cannon and Fenske (R-10) modified the Ostwald so that the upper and lower bulbs lie on the same vertical axis (Figure 4a). For opaque liquids, the viscometer was further modified (R-11) so that the movement of the meniscus could be

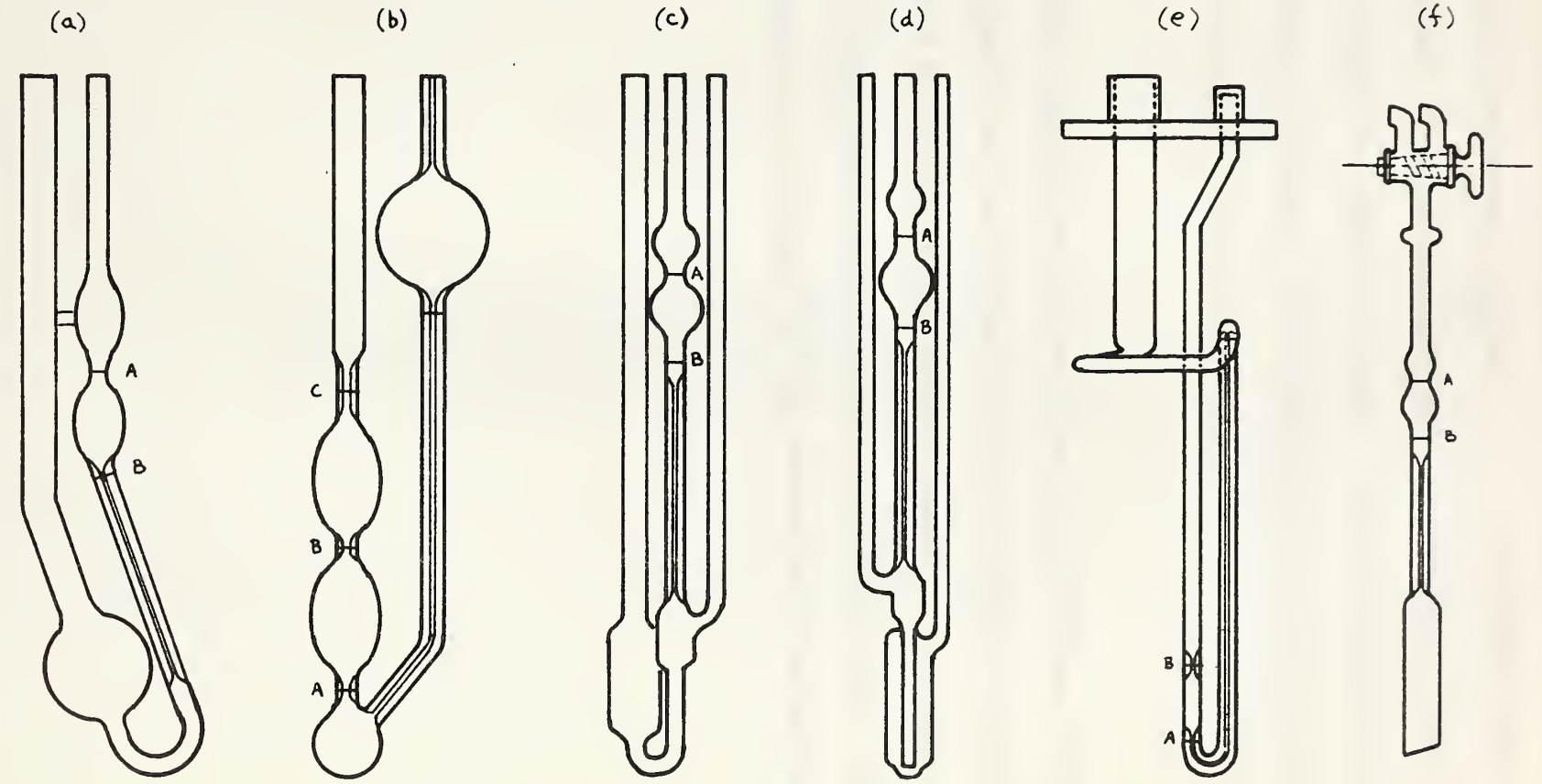
clearly observed as the fluid rose into an initially clean bulb (Figure 4b).

In order to automatically fix the lower liquid level and thus eliminate the necessity of filling the viscometer with a definite volume, Ubbelohde (R-58) introduced the idea of a "suspended level". With this method, the lower end of the capillary widens out into a spherical shoulder so that by introducing an air vent (see Figure 4c) the liquid is induced to flow down the walls of the discharge bulb.

A number of common glass capillary viscometers are shown diagrammatically in Figure 4. The description, calibration, and operation of these and other glass capillary viscometers are extensively described in an ASTM Standard (R-2).

Generally speaking, glass capillary viscometers are not well suited for studying non-Newtonian fluids, primarily because the shear rate varies from zero at the capillary center to a maximum at the wall. In some cases, non-Newtonian fluids may be studied by determining the relationship between the shear stress at the wall of the capillary, $D\Delta P/4L$, and the term $8V/D$. To vary the shear stress, external pressure may often be applied in addition to the hydrostatic head. One instrument specifically designed for studying non-Newtonian

FIGURE 4 COMMON GLASS CAPILLARY VISCOMETERS



CANNON - FENSKE
for Transparent Liquids
for Opaque Liquids

UBBELOHDE

FITZSIMONS

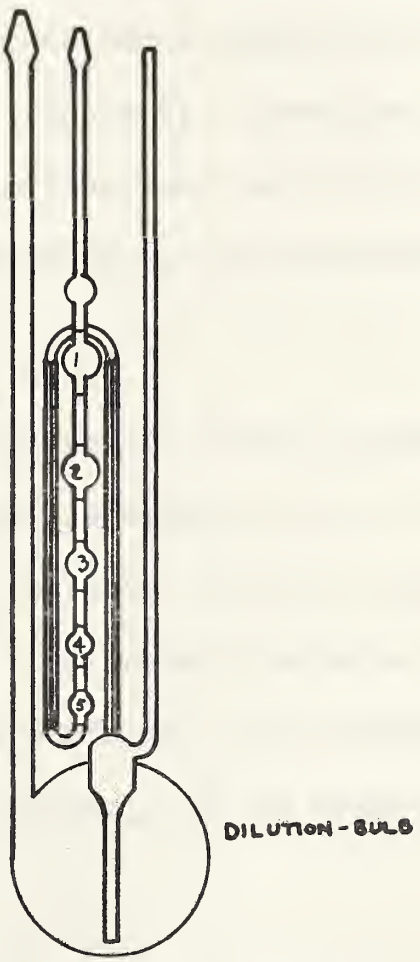
ZEITFUCHS CROSS - ARM

ATLANTIC

fluids has recently been described by Cragg and Van Oene (R-24). This viscometer, Figure 5, is a modified Ubbelohde type viscometer, and is provided with five efflux bulbs, each with its own pair of fiducial marks. With these five bulbs, it is possible to achieve five different accurately reproducible mean heads corresponding to shear stresses varying over a ten-fold range.

Materials exhibiting appreciable yield values, Bingham plastics, should not be studied in glass capillary viscometers as the efflux bulb will never drain properly. In fact, the flow may be completely inhibited by the yield value, since the applied pressure is limited by the mechanical properties of the glass.

FIGURE 5 CRAGG AND VAN OENE VISCOMETER



Orifice Viscometers

Orifice viscometers consist of a short capillary through which a measured amount of sample fluid is discharged. The original design concepts of these instruments were derived from the Hagen-Poiseuille Law. Unfortunately though, the viscometers that were developed consisted of short capillaries or orifices. Under such conditions, the flow does not obey the Hagen-Poiseuille Law. There is, therefore, no simple relationship between the efflux time and the viscosity. As a result this type of viscometer is not suitable for rheological studies.

Even though these instruments cannot be used for studying non-Newtonian fluids, they are widely used in industry, particularly the oil industry, because of their simple operation. The principal types are: the Saybolt Universal (Figure 6) and Saybolt Furol used in North America, the Redwood No. 1 and No. 2 used in Great Britain, and the Engler used in Continental Europe.

Although the design details of these five instruments differ greatly, the method of operation is the same (R-35). The liquid under test is poured into a cup surrounded by a water or oil bath providing proper temperature control. At the base of the cup is fixed a short capillary or orifice with

a simple form of valve. With the level of the liquid in the cup adjusted to a definite height, the valve is opened, and the time required for a stated volume of liquid to discharge through the orifice into a measuring vessel placed below is recorded. This time, generally in seconds, is a purely arbitrary expression of the viscosity.

Because of the complex flow mechanics in this type of instrument, the time readings in the five commonly used instruments are not the same for any given liquid. Conversion formulas or tables (R-5,59) must, therefore, be consulted in order that results may be compared. Even then, such comparisons are only approximate.

Cylinder-Piston Viscometers

The Cylinder-Piston type of viscometer has a cylinder as the liquid reservoir, and the fluid is forced through an orifice or short capillary by a mechanically driven piston or plunger. The mechanism that actuates the piston can be (1) a dead weight, (2) a pneumatic device, (3) hydraulic pressure, or (4) a mechanical device. This type is well suited to studying viscous Newtonian fluids, since the mechanical structure of the viscometers permits the use of very high extrusion pressures.

The Merz-Colwell Extrusion Rheometer (R-36) shown in

FIGURE 6 SAYBOLT VISCOMETER

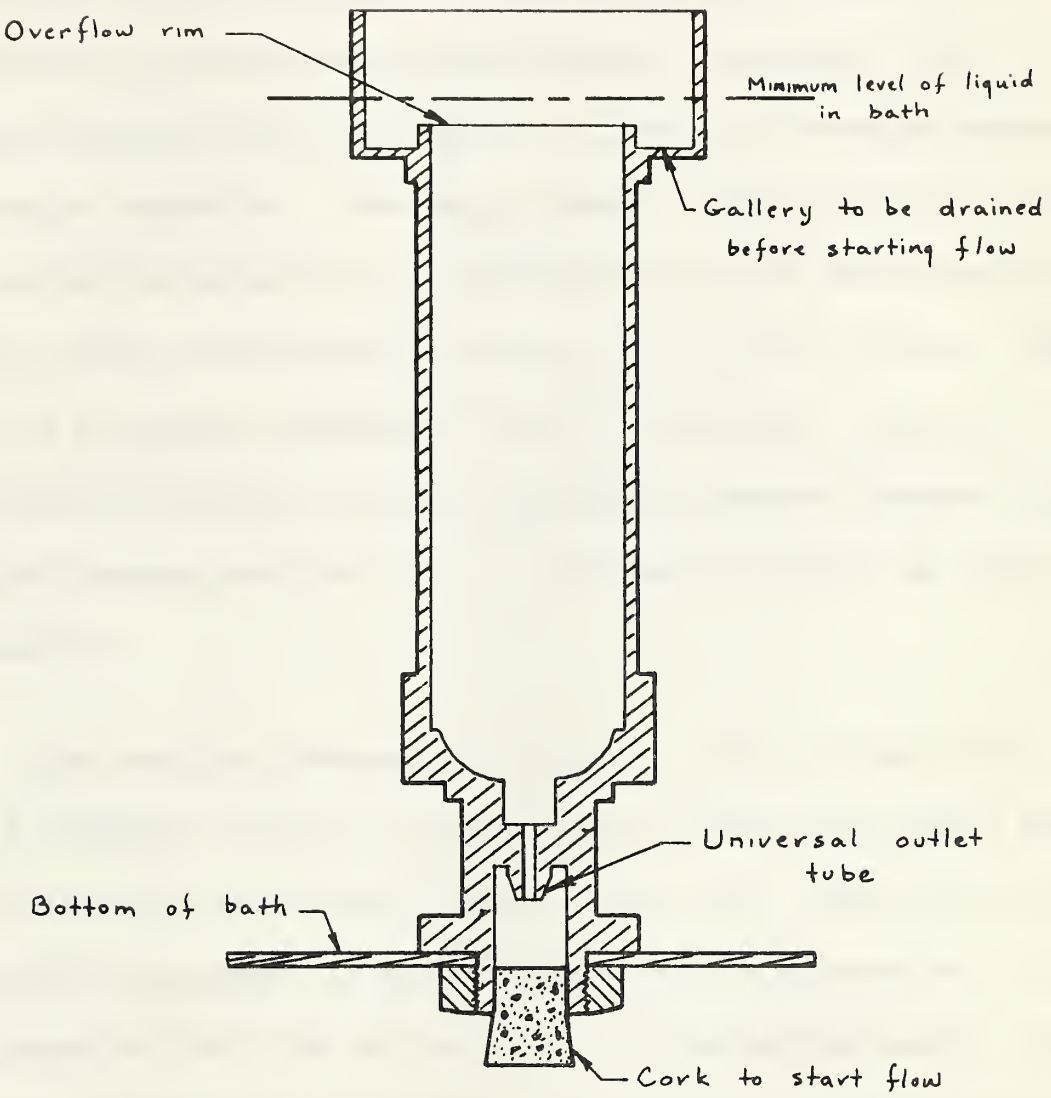
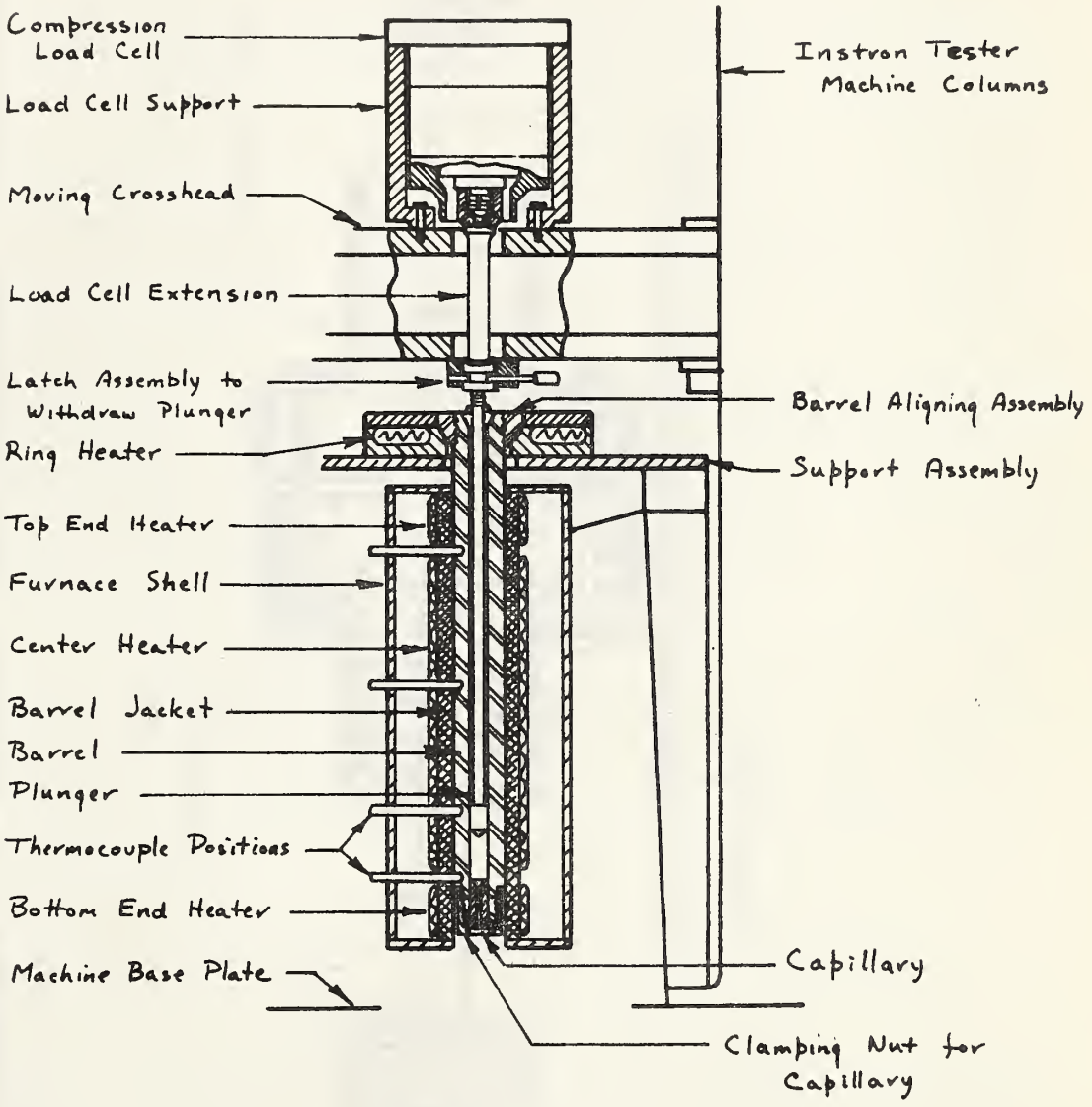

Overflow rim
Minimum level of liquid in bath
Gallery to be drained before starting flow
Universal outlet tube
Bottom of bath
Cork to start flow

Figure 7, is typical of a Cylinder-Piston Viscometer. With this instrument, an Instron Testing Machine is used to drive the piston and cause the test material to flow through the capillary tube. Van Wazer et al (R-60) list a number of other Cylinder-Piston viscometers, namely the Melt Indexer, the Standard Oil Development High Pressure Viscometer, the Burrell-Severs Extrusion Rheometer, and the Pressure Products Industry Rheometer. The Melt Indexer, used extensively to characterize polyethylene and described in an ASTM Standard (R-3), uses a dead weight loading to drive the piston. The SOD is a typical example of the cylinder-piston type of capillary viscometer using a hydraulic-pressure system. The Burrell-Severs and the P. P. I. are both operated by pneumatic pressure.

The Westover Rheometer, Figure 8, first designed by R. F. Westover (R-63) is interesting in that the sample does not leave the instrument during a test run. The test material (usually a polymer) is placed under moderate pressure in the cylinder on one side of the die. As the polymer melts, some of it passes through the die to come in contact with the other ram. After thermal equilibrium has been reached the sample may be subjected to pressures up to 30,000 psi. A differential pressure applied across the die causes flow of the sample through the die. The flow rate is determined from the time

FIGURE 7 MCER CAPILLARY VISCOMETER EXTRUDER ASSEMBLY



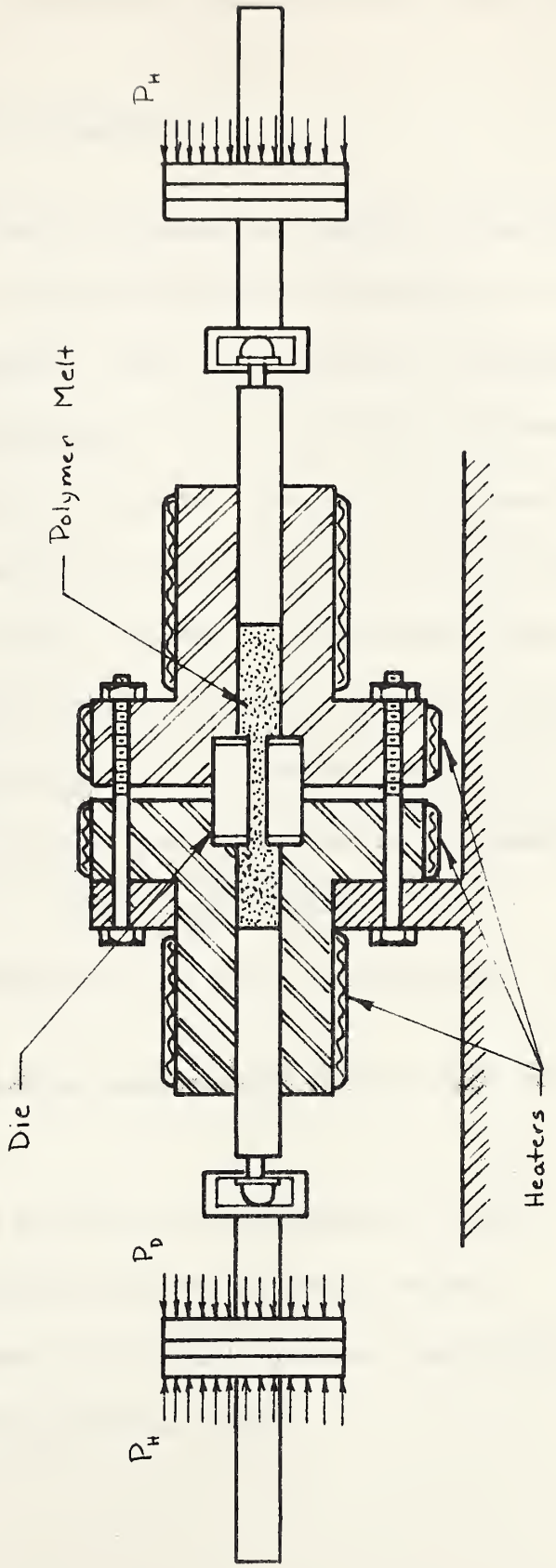


FIGURE 8 WESTOVER HIGH PRESSURE RHEOMETER

required for the ram to travel a specified distance.

Falling-Body Viscometers

Falling-body viscometers include: the falling-ball viscometers, the rolling-ball viscometers, the rising-bubble viscometers, the falling-piston viscometers, and the float viscometers. All of these instruments involve the application of Stokes' Law which relates the viscosity of a Newtonian fluid to the velocity of an object falling through the fluid. Stokes (R-55) showed that if a sphere of radius r and density ρ_s falls through a fluid of density ρ and viscosity μ at a constant velocity v, then the force balance gives the following relationship:

$$\mu = \frac{2r^2(\rho_s - \rho)g}{9v} \qquad (6)$$

where g is the gravity constant, ft/sec^2

The following assumptions (R-40) are made in deriving equation (6):

(1) the motion of the sphere is slow

(2) the fluid is of infinite extent

(3) there is no slip between the fluid and the sphere

(4) the sphere is rigid.

Assumption (1) is satisfied if the Reynolds number, defined as:

$$Re = 2r\rho v/\mu \qquad (7)$$

where r = radius of the sphere

ρ = density of the fluid

v = velocity of the sphere

and μ = viscosity of the fluid

is very much smaller than unity. Assumption (2) obviously cannot be satisfied although it is approximated by using a sphere with a diameter very much less than the diameter of the container. The other assumptions are generally satisfied. Suitable corrections for the effect of a Reynolds number up to two, the wall effect and the tube end effect, are discussed by Oka (R-40) and Merrington (R-35).

Falling-Ball Viscometers

Determination of viscosity by the falling-ball method has long been a standard in industry for relatively viscous and transparent solutions. The method is extremely simple and consists of measuring the time required for a glass or steel ball to fall a measured distance through the sample fluid contained in a vertical glass tube whose diameter is relatively large compared to the ball.

A falling-ball viscometer may be made from a large graduated cylinder with a rubber stopper having a centered glass tube in it for insertion of the balls. The time required for suitably chosen balls to fall through the test fluid is then determined after a constant steady-state velocity has developed. For opaque liquids, the movement of a metal ball past a fixed point can be sensed electrically (R-60). Such a detection system, used in the Fritz and Weber Viscometer (R-18), consists of several turns of wire about the cylinder at the correct point. This coil of wire then acts as the inductance in a resonating electrical circuit which is detuned by the passage of the metal ball through the coil.

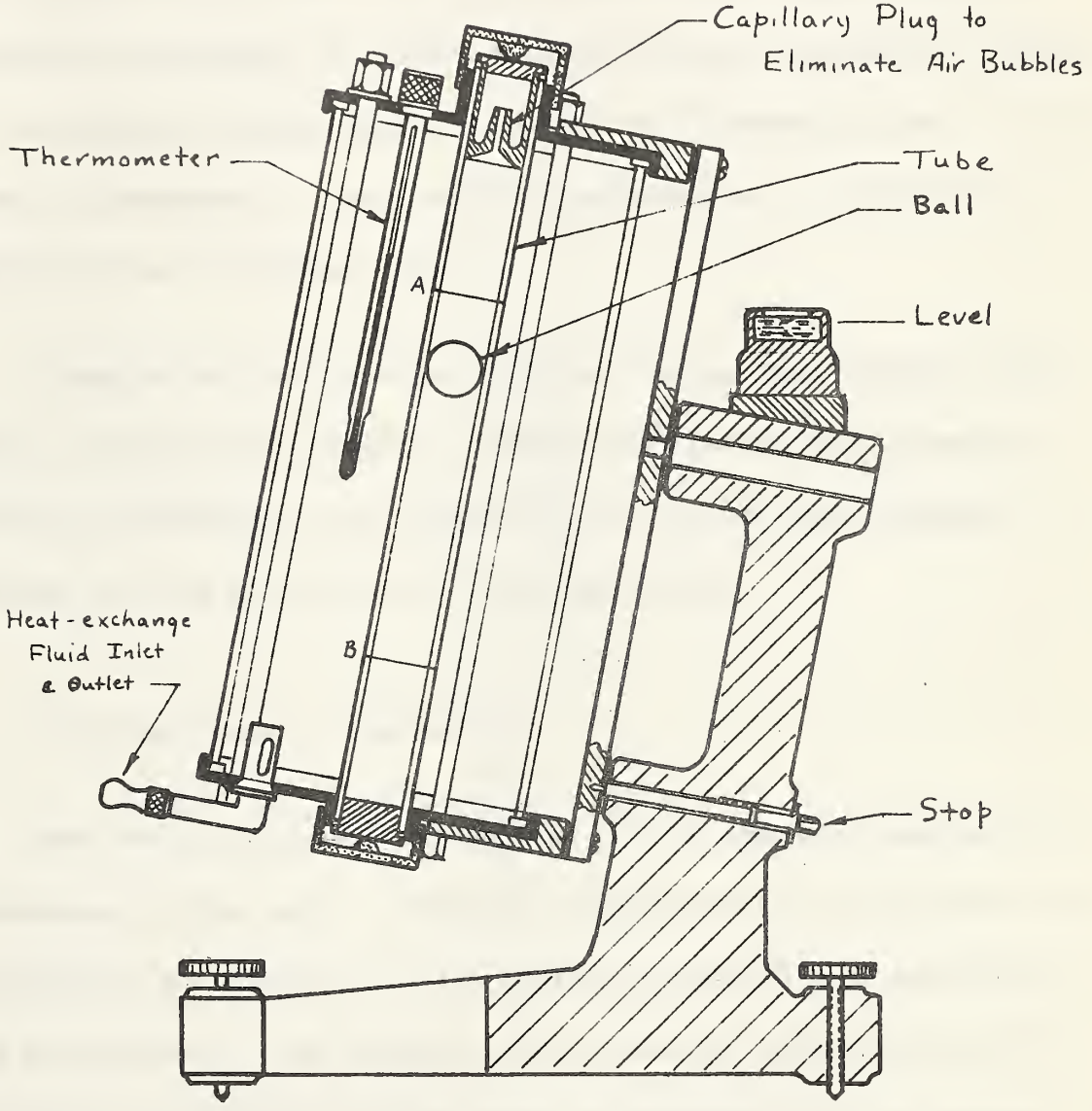
Rolling-Ball Viscometers

The rolling-ball viscometers are similar to the falling-ball viscometers in that the speed with which a sphere rolls down a cylindrical tube, filled with a fluid and inclined at a fixed angle to the horizontal, gives a measure of the viscosity of the fluid. The mathematics of this case are quite complicated, however, Hubbard and Brown (R-31), by dimensional analysis, have developed an equation relating the instrument dimensions, the velocity of the ball, and the viscosity of the fluid.

A schematic diagram of the Hoeppler Rolling-Ball Viscometer is shown in Figure 9. This instrument is extremely easy to operate and permits any number of check runs by simply rotating the inclined tube on its support. The time required for the ball to roll between the graduated marks on the tube is recorded as a measure of the fluid viscosity.

A very elaborate instrument of the rolling-ball type is the Ruska Viscometer which is designed for operation at high pressures and high temperatures. This commercially available instrument is applicable to Newtonian fluids only.

FIGURE 9 HOEPPLER ROLLING-BALL VISCOMETER



Rising-Bubble Viscometers

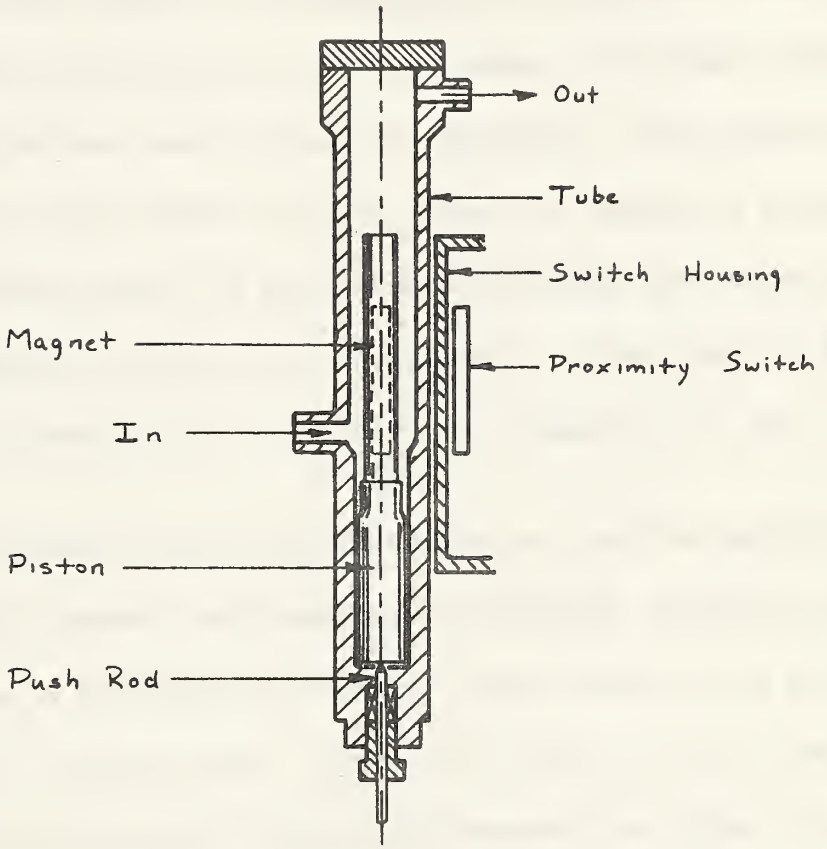
The rise of an air bubble through a liquid medium affords a simple visual measurement of the fluid viscosity. Although the rheological theory of rising gas bubbles is not well understood, Bond (R-9), by making some simplifying assumptions regarding the motion of the fluid inside the bubble, has managed to formulate a mathematical expression for a rising-bubble viscometer. The resulting expression is actually a modification of Stokes' Law.

Commercial instruments such as the Gardner Bubble Viscometer and the Interchemical Bubble Viscometer are commonly used for estimating the viscosity of varnish and lacquers because of the simplicity of the operation.

Falling-Piston Viscometers

The falling-piston viscometer is an absolute method for Newtonian fluids only. Relative measurements can be made for non-Newtonian fluids only if rigidly standardized conditions are maintained. The method, first used by Pochettino (R-42), consists of determining the rate of movement of the piston within an outer cylinder both of which are submerged in the material to be tested. Normally the outer cylinder remains stationary and the piston moves axially while the fluid to be

FIGURE 10 NORCROSS VISCOMETER



tested occupies the annular space between the cylinders.

The Norcross Viscometer (R-60), shown in Figure 10, is a commercially available instrument that uses the falling-piston principle. The instrument is designed for continuous operation in viscosity process-control. It is operated by a push rod which periodically raises the piston and allows a sample of the liquid to be measured to be drawn into the space formed below the piston as it is raised. The push rod is then quickly withdrawn and a timer is started. The piston falls under gravity and shuts off the timer by means of a proximity switch located near the end of the cylinder and operated by a magnet embedded in the piston assembly. The time of fall, which is automatically recorded, is a measure of the viscosity.

Penetrometers are modifications of the Pochettino Viscometer. With these instruments, a cylinder, needle or cone is pushed into a viscous fluid by a given force, to a given depth, and/or for a given time. Since the piston is not completely submerged in the sample as in the Pochettino type, the piston tends to cause a large displacement of the test liquid in the opposite direction to the piston motion. The mathematical relationships for these instruments have been derived (R-53), for Newtonian fluids.

The Precision Universal Penetrometer is an industrial instrument made by the Precision Scientific Company and is used for testing greases, jams, marmalades, and paints.

A Mobilometer is similar to a Penetrometer except that the plunger is a perforated plate, and the liquid under test is squeezed through the perforations as the plunger descends in the tube. The time taken for the plunger to descend is a measure of the viscosity. The Gardner Mobilometer (R-19) and the SIL Mobilometer are two commercially available instruments.

Float Viscometers

The Float Viscometer is an adaptation of the rotameter-type flow meter. The instrument consists of a vertical tapered tube and a float that moves within it. The float is designed such that, for a constant flow rate, the elevation of the float is a measure of the fluid viscosity.

Obviously the maintainance of a constant flow rate is of the utmost importance with these instruments. A constant flow rate is often maintained by a positive displacement metering pump or, less precisely, by a differential flow regulator.

The Fisher and Porter Viscorator and Continuous Plunger

Viscometer are commercially available float viscometers.

Rotational Viscometers

Rotational viscometer designs are based on the fact that a rotating body, immersed in a fluid, experiences a viscous drag or retarding force. From the relationship between the angular velocity and some measure of the retarding force, the viscosity of the fluid can be calculated. The main advantage of rotational viscometric procedures is that the rotational instrument provides a nearly constant shear rate throughout the entire fluid. Furthermore, a sample may be subjected to a given rate of shear or shear stress over an extended period of time. With some rotational viscometers, it is also possible to vary the rate of shear over a wide range. Because of their attributes, rotational viscometers are well suited to the study of non-Newtonian fluids and have become the most widely used class of instruments for rheological determinations.

Originally rotational viscometers were coaxial cylinders which resembled a shaft and bearing and sometimes a small mixing tank (R-6). Today, besides the concentric cylinder system, there are many variations including a disc, paddle, prong, or plate and cone arrangement.

Concentric Cylinder System

Rotational viscometers, in which the material to be tested is contained between two vertical coaxial cylinders, fall into one of two general types: (1) one cylinder is rotated at a definite velocity and the torque required to prevent the other cylinder from rotating is measured, and (2) one cylinder is held stationary and the velocity of rotation of the other cylinder under a fixed torque is measured.

In order to arrive at the fundamental equations for the concentric cylinder system, the following assumptions are made (R-40):

(1) the fluid is incompressible

(2) the motion of the fluid is laminar

(3) the streamlines of flow are circles on the horizontal planes perpendicular to the axis of rotation, ie. centrifugal forces are neglected

(4) the flow is steady

(5) there is no slippage at the wall of either cylinder

(6) the motion is two dimensional, ie. end effects are neglected

(7) the system is isothermal.

To derive the basic equations for a coaxial cylinder viscometer, suppose that a cylinder of radius R_b is suspended

within a larger cylinder of radius R_c. Suppose also that the outer cylinder is rotated at a constant angular velocity Ω, and that the fluid in the space between the cylinders is time-independent and can be described by the simple rheological equation:

$$\dot{\gamma} = f(\tau) \tag{8}$$

where $\dot{\gamma}$ = rate of shear

τ = shear stress

At any radius R, in the annular space between the cylinders, the shear stress is given by:

$$\tau(R) = \frac{G}{2\pi R^2 h}$$

where G is the measured torque restraining the centre cylinder

h is the depth of immersion of the center cylinder

and the rate of shear is given by:

$$\dot{\gamma} = R \frac{d\omega}{dR}$$

where ω is the angular velocity at radius R.

substituting in equation (8) gives:

$$R \frac{d\omega}{dR} = f\left(\frac{G}{2\pi R^2 h}\right)$$

Integration between $\omega = 0$ at $R = R_b$ to $\omega = \Omega$ at $R = R_c$ gives:

$$\Omega = \int_{R_b}^{R_c} f\left(\frac{G}{2\pi R^2 h}\right) \frac{dR}{R} \tag{9}$$

Substituting $\tau(R) = \frac{G}{2\pi R^2 h}$ and $\frac{dR}{R} = -\frac{d\tau}{2\tau}$ in (9) gives:

$$\Omega = \int_{\tau_c}^{\tau_b} \frac{f(\tau)\, d\tau}{2\tau} \tag{10}$$

For a Newtonian fluid where

$$f(\tau) = \frac{\tau}{\mu} \text{ and } \tau = \frac{G}{2\pi R^2 h}$$

thus equation (10) becomes:

$$\Omega = \int_{\tau_c}^{\tau_b} \frac{d\tau}{2\mu} = \frac{\tau_b - \tau_c}{2\mu}$$

and since $\tau_b = \frac{G}{2\pi R_b^2 h}$

and $\tau_c = \frac{G}{2\pi R_c^2 h}$

Hence, after rearranging:

$$\mu = \frac{(R_c^2 - R_b^2)\, G}{4\pi h \Omega R_c^2 R_b^2} \qquad (11)$$

Equation (11) is the well known Margules Equation for the flow of a Newtonian fluid in a rotational viscometer. The important Krieger and Maron (R-32) equation and other similar equations for non-Newtonian fluids are discussed by Oka (R-40), Wilkinson (R-64) and Van Wazer et al (R-60).

Equation (11) is unchanged if, in the derivation, the inner cylinder rather than the outer cylinder is assumed to be rotating. On the other hand, the criteria for flow stability in the viscometer annulus is very much dependent on which cylinder is rotating. The usual criteria for flow stability is that the Reynolds number be less than some critical value. For a rotational viscometer, the Reynolds number is given as (R-50a):

$$Re = \frac{2\pi S R (R_c - R_b) \rho}{\mu} \qquad (12)$$

where S = speed of the rotation cylinder RPS

R = radius of the rotating cylinder

R_b = radius of the inner cylinder

R_c = radius of the outer cylinder

ρ = density of the test fluid

μ = viscosity of the test fluid.

Schlichting (R-50a) reports that the transition from laminar to turbulent flow in the annulus between concentric cylinders is influenced by centrifugal forces. When the outer cylinder is at rest, and the inner cylinder is rotating, the fluid particles near the inner wall experience a higher centrifugal force and show a tendency to be propelled outward thus resulting in flow instability. This flow instability takes the form of circular flow patterns within sections of the annulus. The critical Reynolds number, above which these so called "Taylor vortices" occur, is given by:

$$Re_{crit.} = 41.3 \sqrt{\frac{R_c}{R_c - R_b}} \qquad (13)$$

When the inner cylinder is stationary and the outer cylinder is rotated, the inertial forces have a highly stabilizing effect on the flow. In this case the critical Reynolds number, above which turbulent flow occurs, is much higher than the value given by equation (13) and is usually taken as:

$$Re_{crit} \simeq 3500 \qquad (14)$$

In deriving equation (9) the end effects of the cylinders

were neglected. In practice of course, the cylinder ends are also subjected to a viscous drag, therefore, this effect must be eliminated if equation (9) is to apply. Normally the end-effect may be considered as equivalent to an increase in the effective depth of immersion, h. Although the end-effect may be calculated from the instrument geometry (R-49), it is often better to obtain the end correction experimentally. The angular velocity, Ω, and torque, G, at several immersion depths, h, are plotted as G/Ω versus h. The plot is extrapolated, and the negative of the intercept on the h axis is the end correction to be added to the effective depth of immersion used in equation (9).

Mooney and Ewart (R-38) have shown that the concentric cylinder system may be modified in such a manner that the viscous drag on the bottom of the inner cylinder may be calculated, thus eliminating the end effect altogether. With their modification the lower ends of both cylinders are tapered to form cones, having a common apex which forms the lower bearing point of the inner cylinder. The cone angles are chosen so that the ratio of the clearance at any point to the radial distance from the axis is constant and approximately equal to the ratio of the clearance to the radial distance for the two cylinders. This ensures that the velocity gradient is essentially constant throughout the material. The viscosity may then

be calculated using a modified form of equation (10).

For any particular apparatus, equation (11) can be reduced to the form:

$$\mu = \frac{G}{N} \times \text{constant} \qquad (15)$$

Thus, if a rotational viscometer is to be used routinely on fluids that differ only slightly in their flow properties, the calculations of the viscosities may be simplified by determining the instrument constant. The constant is obtained simply by calibrating the instrument with a standard fluid whose viscosity is known for the conditions under study.

For studying time-dependent effects, the rotational viscometers, with a very small clearance between the cup and bob, are particularly well suited. They enable the determination of the complete shear stress-shear rate curve, and they permit a study of the progressive decrease (or increase) in torque as time increases at constant rotational speeds.

Rotating Cup Viscometers

The Couette Viscometer (R-12) is one of the earliest coaxial cylinder instruments using a rotating outer cylinder and a suspended inner cylinder. Hatschek's (R-29,30) modification of the Couette apparatus is shown in Figure 11. With these instruments, the inner cylinder is suspended from

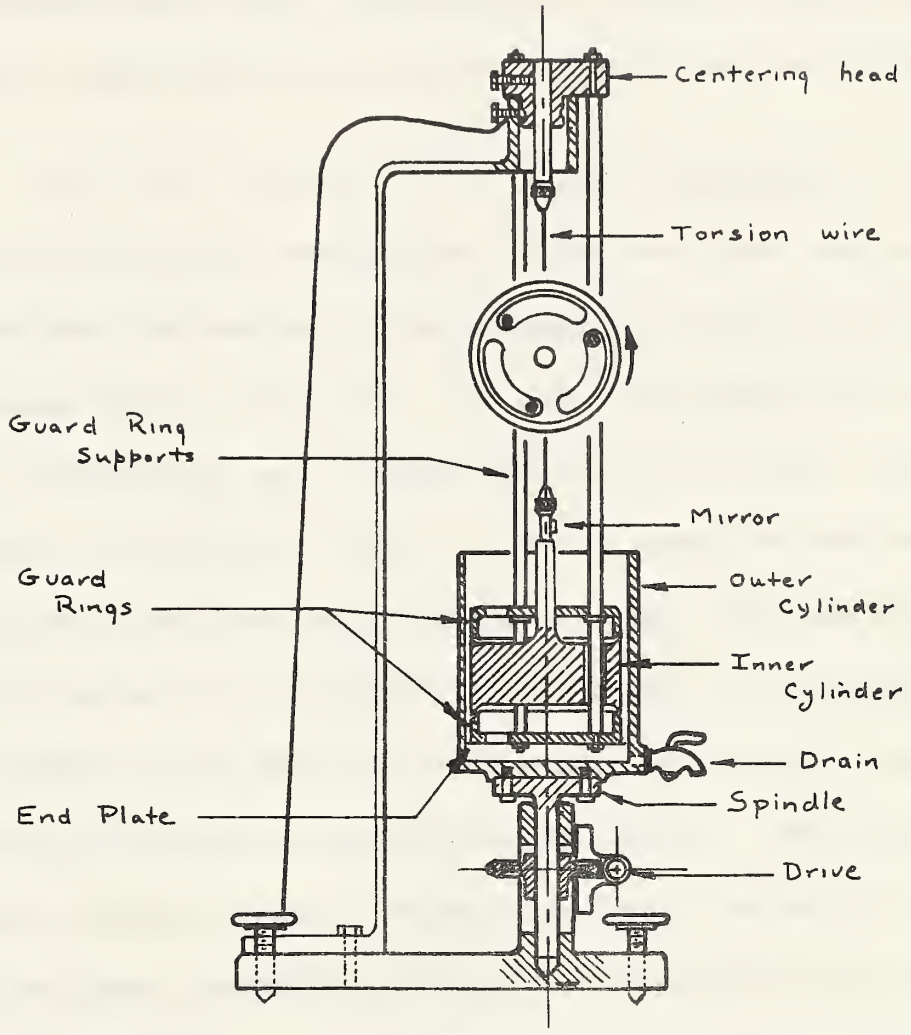
a torsion wire, and the outer cylinder rests on a spindle and is rotated at a constant speed. The deflection of the center cylinder is read by a mirror with scale and telescope. Guard rings leaving only a small clearance between themselves and the suspended inner cylinder, are used to eliminate the end effects. An end plate having a diameter only slightly smaller than the outside cylinder provides further protection against the effect of the bottom.

The MacMichael Viscometer (R-23) also uses a torsion wire to support the inner cylinder. With this instrument the end-effects are found experimentally by using different depths of sample fluid. Two drawbacks of the instrument are the limited range of speed of rotation of the cup, and the difficulty of maintaining accurate temperature control.

A unique torque-sensing system is employed by the Agfa Rotational Viscometer manufactured by Agfa Aktiengesellschaft of Munich, Germany. The inner cylinder is attached to a rigid torsion rod which is fitted with arms or lateral extensions of the rod. The movement of these arms, which gives a measure of the restraining torque, is detected by a linear differential transformer.

Green (R-26) designed a viscometer, now available from the Precision Scientific Company, that is constructed in such

FIGURE 11 HATSCHEK'S CONCENTRIC CYLINDER APPARATUS



a way that the cup speed can be increased or decreased in small steps over a wide range. The viscous force exerted on the bob is measured by a helical torsion spring which supports the bob.

The Fann V-G Meter is also similar in principle to the MacMichael and Agfa instruments. In this case the torque on the inner cylinder is measured by a torsion spring.

The novel feature of the Polard Viscometer is the virtually frictionless suspension of the bob, and the totally friction-free method of determining the torque set up by the viscous drag on the bob. The bob has conical ends to reduce the end-effects and is weighted so that it will just barely float in the sample fluid. A shaft from the bob meets a jeweled pivot bearing at the top, which will be essentially frictionless if the buoyancy of the bob is adjusted properly. Attached to the bob-shaft are vanes which turn, with the motion of the bob, between stator plates. The vanes and stator plates act as a capacitor when a potential is applied. The voltage required to bring the vanes to a null point for various shear rates is a measure of the shear stress.

Goodeve (R-21) described a modification of the Couette Viscometer in which the concentric cylinders are replaced by two concentric cones. The main advantage of the arrangement

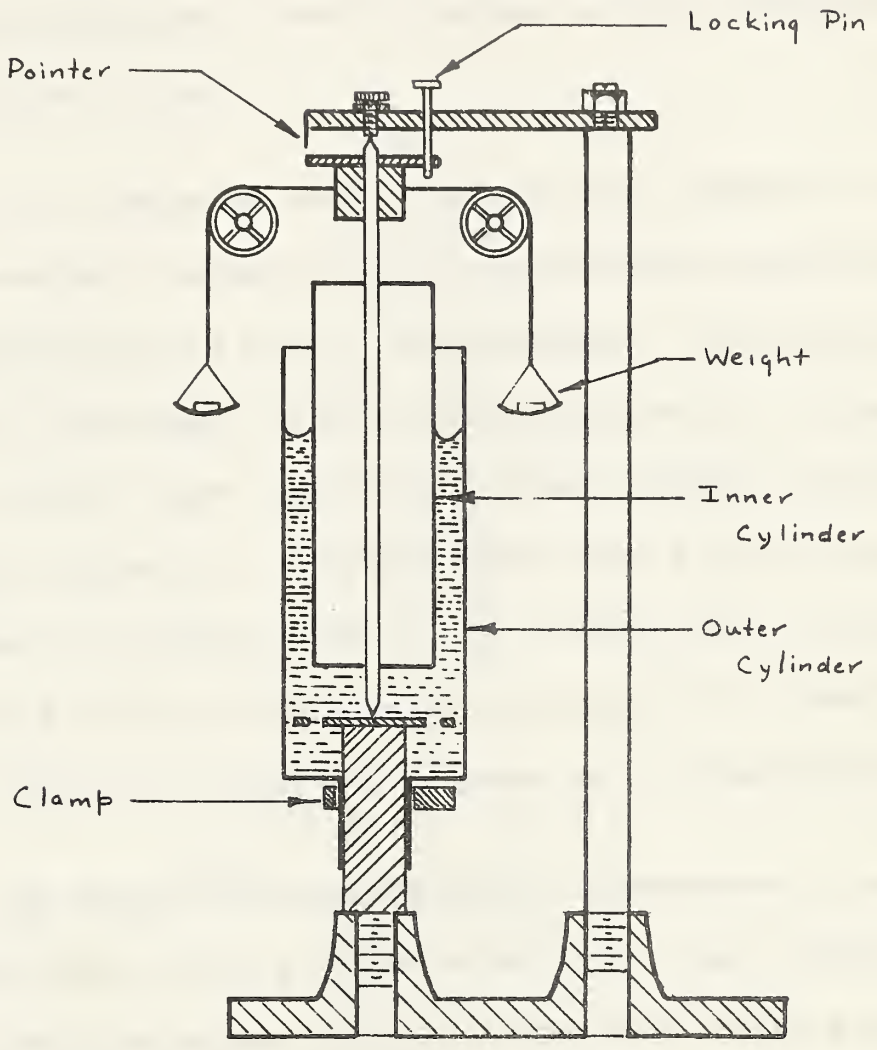
is that the rate of shear can be conveniently altered by moving one cone along the axis, thus altering the separation between the opposing conical surfaces. The instrument has been used for studying thixotropic behavior of some suspensions.

The Alberta Rotational Viscometer used for a number of years at the University of Alberta and described by Ritter (R-46) operates on the same principle as the MacMichael Viscometer. However, unlike the MacMichael Viscometer, the Alberta instrument has been specially designed to provide accurate temperature control and a wide range of cup speeds. Efforts have also been made to eliminate end-effects.

Rotating Bob Viscometers

The earliest instrument to operate on the principle of a fixed outer cylinder and a rotating inner cylinder was Searle's apparatus (R-51). The apparatus (Figure 12) consists of an inner cylinder carried by a spindle working in fixed bearings, the lower one being located in the top of the pillar which supports the outer cylinder. A constant torque is applied by means of two weights carried by threads wound around a drum on the inner cylinder axle and passing over ball-bearing pulleys. The time of one revolution of the inner cylinder is determined. A locking pin enables the inner cylinder to be stopped.

FIGURE 12 SEARLE'S CONCENTRIC CYLINDER APPARATUS

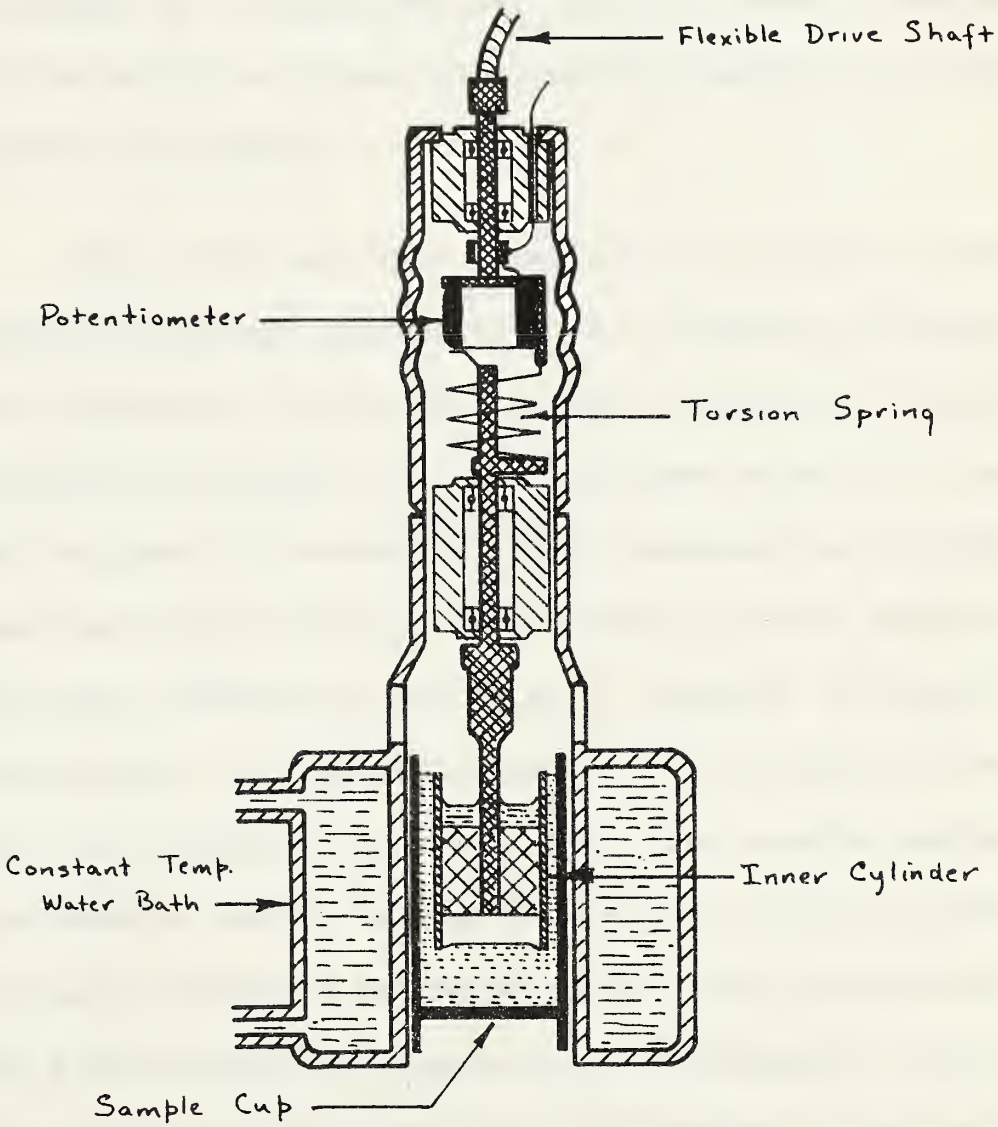


The commonly used Stormer Viscometer (R-56) operates on the same principle as the Searle. The velocity of the rotating cylinder, actuated by a weight, is measured by a revolution counter attached to the spindle. Various types of rotors such as cylinders, fork paddles, and propeller paddles are used although absolute viscosity values are best obtained with a cylindrical rotor.

It is reported (R-60) that the most versatile viscometer now available commercially is the Rotovisco manufactured by Gebruder-Haake in Berlin, West Germany. The measuring head of this instrument is shown diagrammatically in Figure 13. The rotating inner cylinder is driven through a precision torsion spring by a flexible shaft from a synchronous motor located in a control panel. The viscous drag on the rotor is measured by the deflection or twisting of the torsion spring. This twisting is measured by means of a potentiometer.

The Brookfield Synchro-Lectric Viscometer is especially adapted for use as a portable machine. When a spindle rotating at a definite constant speed is immersed in the sample fluid, the viscous drag opposing the motor is indicated on a rotating dial by means of a pointer and is expressed in terms of viscosity. The pointer and one end of a spiral spring are attached to the spindle, while the other end of the spring is attached to the dial which in turn is directly connected

FIGURE 13 HAAKE ROTOVISCO VISCOMETER MEASURING HEAD



to the constant speed motor.

Similar instruments, designed for the continuous measuring of viscosity in process lines, are the Brookfield Viscometran and the Brookfield Viscosel. In the Viscometran, the torque of the spring is transmitted by either a variable condenser or a potentiometer. In the Viscosel, the torque of the spring is converted to an air pressure by a flapper-nozzle arrangements.

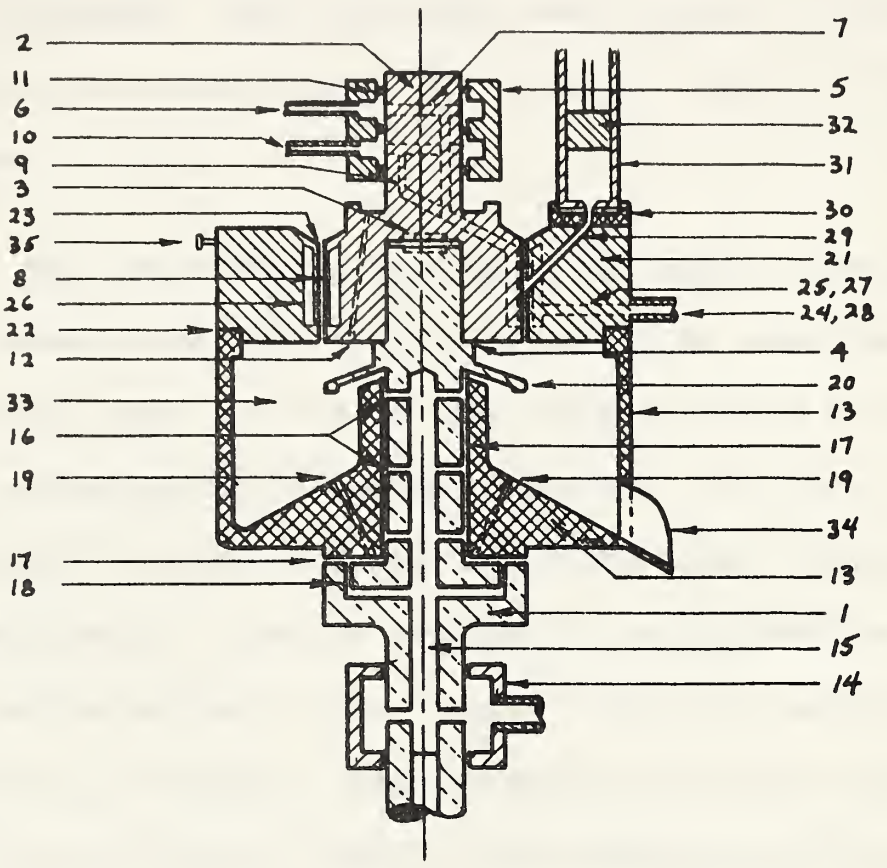
There are also other commercially available viscometers which utilize the viscous drag on a constantly rotating spindle as a measure of viscosity. Amongst these are: the Corn Industries Viscometer built by Gaertner Scientific Company and designed for measuring starch suspensions; the Hallikainen-Dow Viscometer designed for continuous process analysis; and the Drage Rheometer built by A. G. Epprecht in Zurich, Switzerland; and the Visco-Corder or Visco-Amylo-Graph built by C. W. Brabender Instruments Inc. for routine testing. Instruments similar to the Farinograph also use the same viscosity measuring technique as the above instruments. The Farinograph manufactured by C. W. Brabender Instruments Inc. is designed for studying flour doughs. The instrument consists of a mixer or kneeding head driven at a constant speed by a motor which is free to turn around its shaft in

order to lift a lever attached to a spring balance. As the torque on the motor increases, the lever rises giving a measure of the dough properties.

Two very interesting viscometers in which the inner cylinder rotates are the Merrill-Brookfield Viscometer and the Hercules High Shear Viscometer. In both these instruments the shear stress is measured at the wall of the outer cylinder rather than at the surface of the inner cylinder.

The Merrill-Brookfield Viscometer (R-34) is unique for a number of reasons. As shown in Figure 14, the drive shaft (1) serves to drive the rotor, to act as the vertical axis of the measuring head, and to distribute air to the air bearing. The non-rotating frame (13) is carried on the shaft and separated from it by a thin film of air which covers all the surfaces between the frame and drive shaft. The top of the air bearing is protected from fluid from above by the umbrella-like projection (20) which is part of the drive shaft. The fluid to be tested is injected into the shearing annulus (23) which is about 0.006 inches wide and 1.0 inches high. End-effects have been eliminated since there is no bottom to the cup. The temperature of both the stator and the rotor may be controlled by circulating a heat exchange fluid through the channels provided. (7,8,9,24,25,26,27 and 28).

FIGURE 14 MERRILL-BROOKFIELD VISCOMETER MEASURING HEAD



KEY : 1 = Drive Shaft; 2 = Rotor; 3 = Key; 4 = Shoulder; 5 = Stationary Manifold; 6 = Inlet from Thermostatic Bath; 7 = Internal Holes for Heat-exchange Fluid; 8 = Circumferential Channel in Rotor; 9 = Internal Exit Holes for Heat-exchange Fluid; 10 = Exit Tube for Heat-exchange Fluid; 11 = O-rings; 12 = "Weep Hole"; 13 = Nonrotating Frame; 14 = Nonrotating Manifold for Air to Bearing; 15 = Central Bore in Shaft; 16 = Holes for Air to Bearing; 17 = Bearing Surface; 18 = Vertical Holes to Bearing; 19 = Air Relief Holes; 20 = Projection to Protect Air Bearing; 21 = Stator; 22 = Shoulder of Frame; 23 = Shearing Annulus; 24 = Heat-exchange Fluid Inlet Tube; 25, 27 = Heat-exchange Fluid Exit; 26 = Channel in Stator; 28 = Outlet Tube; 29 = Inlet Hole for Introducing Test Sample; 30 = Threaded Base for Syringe; 31 = Syringe; 32 = Syringe Piston; 33 = Chamber for Catching Fluids; 34 = Waste Spout; 35 = Pin for Linkage to Transducer.

The shear stress on the stator is detected by means of a transducer attached to the outer surface of the stator. The output of the strain gauges in the transducer is fed to a recorder. The instrument should prove a valuable analytical tool particularly for studying effects of high shear rates.

With the Hercules High Shear Viscometer, the sample is placed in the cup and the viscous drag caused by the constant speed rotation of the rotor is sensed by means of two coiled springs attached to the cup. The cup is set in bearings to permit essentially frictionless rotation without eccentricity. A pen is attached to the cup wall so that as the cup moves due to the viscous torque, the pen makes a trace on a recorder. This instrument is fairly inexpensive and can be used for studying time-dependent effects if temperature control is not required.

Cone and Plate Viscometers

In the cone and plate viscometer a cone with a large obtuse angle is placed on a flat plate. The apex of the cone just touches the plate surface and the fluid to be tested fills the narrow gap formed by the cone and plate. One of the surfaces is fixed; the other rotates around the axis of the cone. If edge-effects are neglected it can be

shown (R-40,64) that, if the angle between the plate and the cone is less than about 3^{o}, the rate of shear across the conical gap may be considered constant. This means that the viscosity of a Newtonian fluid will be given by:

$$\mu = \frac{3 \alpha G}{2 \pi R^3 \Omega} \qquad (16)$$

where μ = the absolute viscosity

α = angle between plate and cone

G = torque

R = radius of cone

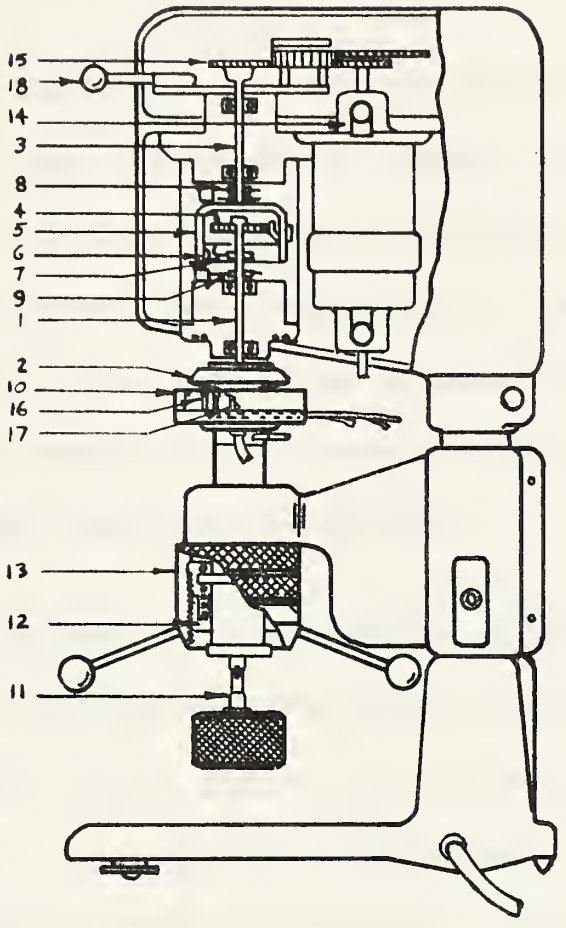
Ω = angular velocity.

Alternately the flow curve may be constructed by plotting the rate of shear (Ω/α) against the corresponding shear stress ($3G/2\pi R^3$).

Either the cone or the plate may be rotated while the other is held stationary. In the Ferranti-Shirley Cone-Plate Viscometer, shown schematically in Figure 15, the flat plate (lower plate) is held stationary while the cone is rotated at a constant speed by a variable speed motor through a gear train and torsion spring. The torque due to the viscous drag on the cone is measured by a potentiometer on the spring which sends a signal to a recorder.

The bottom plate can be raised or lowered and is provided with an electrical connection that indicates on a control cabinet when the plate is just touching the apex of

FIGURE 15 FERRANTI-SHIRLEY PLATE AND CONE VISCOMETER



KEY: 1.= Cone Spindle; 2 = Cone; 3 = Driving Spindle; 4 = Torque Spring; 5 = Bridge Housing; 6 = Potentiometer, for Torque Dynamometer; 7 = Wiper for Potentiometer; 8 = Slip Rings; 9 = Slip Ring; 10 = Plate; 11 = Micrometer; 12 = Nut; 13 = Screw, for Raising Plate; 14 = Driving Motor; 15 = Gearing; 16 = Thermocouple; 17 = Water Jacket; 18 = Gear Shift having a Neutral Position and Two Speeds.

the cone. This setting is achieved reproducibly by means of a micrometer adjustment in the base. The plate is also fitted with a cooling system and supports three thermocouples which are in direct contact with the test fluids in the gap.

With the Rheogoniometer, manufactured by Farol Research Engineers Ltd., Bognor Regis, Sussex, England, the lower plate is driven at a constant speed and the upper cone is held stationary. The torque that is transmitted through the test fluid to the upper cone is measured by the deflection of a rigid torsion rod in much the same manner as was used in the Agfa Rotational Viscometer.

A null-point servo-mechanism is provided to maintain a constant gap between the plate and cone, while the thrust on the lower plate, due to the normal force established in the fluid, is detected by a transducer. This unique feature of the Rheogoniometer means that it is well suited for studying non-Newtonian flow systems, particularly those which exhibit the Weissenberg effect.

Vibrational Viscometers

One instrument is available that determines the viscosity of the test fluid from the viscous dampening of a vibrating blade. This instrument, the Bendix Ultra-Viscoson,

consists of (1) a probe bearing a blade that vibrates at an ultrasonic frequency and (2) the accessory electronic equipment for measuring the viscous dampening of the blade. The flow properties of the fluid are expressed on a meter in terms of the apparent viscosity times the fluid density that is in $gm^2/sec\ cm^4$.

The instrument has been subjected to a detailed mathematical analysis by its designers (R-48). It is not well suited for studying the rheological properties of non-Newtonian fluids since it is essentially a single point instrument from which one obtains an apparent viscosity corresponding to some unspecified average rate of shear. Extreme care must be taken, when using the instrument, to prevent any phase changes in the test sample, since any deposits or bubbles forming around the vibrating blade will cause large changes in the readings obtained.

During recent years, the viscoelastic behavior of plastics and polymer solutions has received considerable attention. As a result, several elastometers (R-17,24,61) have been developed in which the test material is subjected to a sinusoidal oscillation. Although there are no commercially available oscillatory viscometers, a number of the rotational viscometers can be modified for the measurement of viscoelastic properties. The measurement of viscoelasti-

city is discussed by Van Wazer et al (R-60) and by Ferry (R-17).

Parallel-Plate Viscometers

For materials having very high viscosities such as tars, rubbers and plastics, the parallel plate viscometer is probably best suited. With these instruments, the material to be tested is placed between parallel circular plates, and a constant force is applied perpendicular to the plates to cause them to approach each other. From the displacement of one plate with respect to the other as a function of time, the rheological behavior of the sample may be determined.

The equations necessary to reduce the plate separation-time data to fundamental quantities have been developed for Newtonian fluids (R-15), for some viscoelastic bodies (R-14) and for a few other non-Newtonian fluids (R-49).

Sliding Plate Viscometers

The simple arrangement of parallel sliding plates offers a convenient way of measuring fluid properties. The method is equally applicable to materials having Newtonian or certain non-Newtonian flow properties, providing the yield values are not too high. Because of the simple geometry of

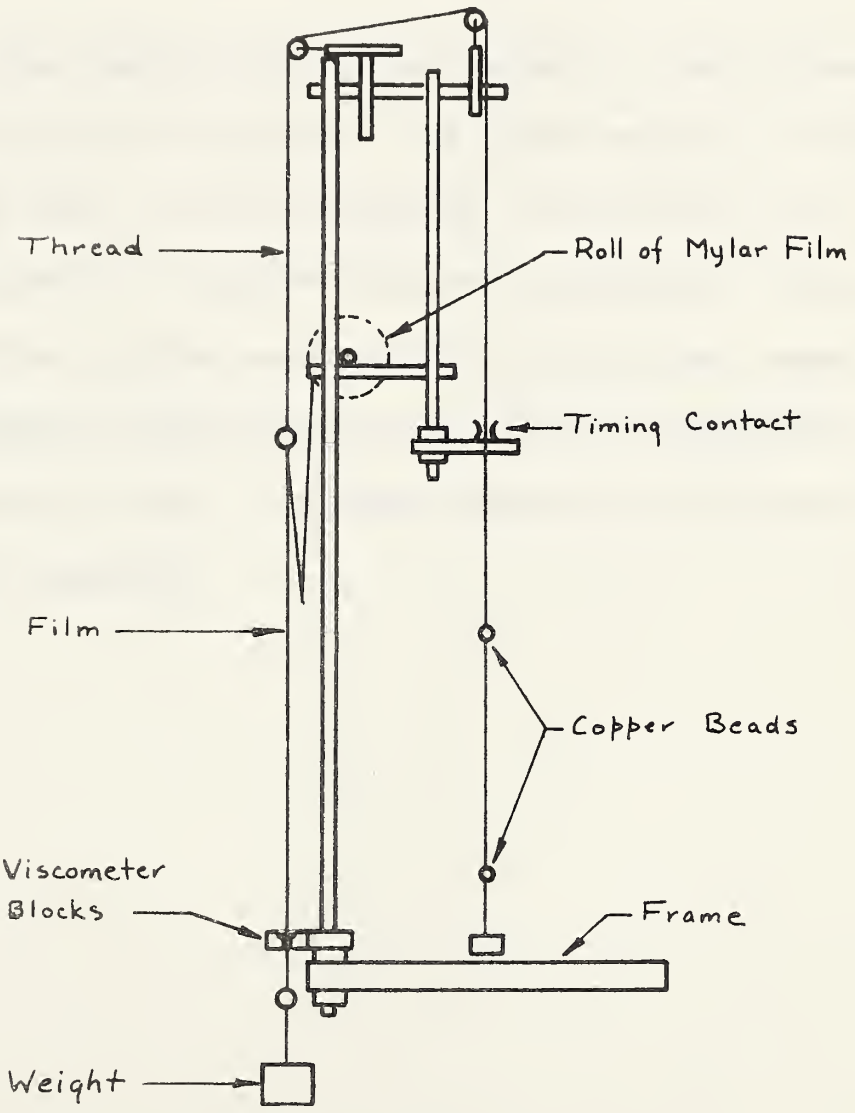
the parallel plate instruments, the shear stress and shear rates can be readily determined (R-60), thus giving the viscosity in absolute units.

There are three generally used arrangements for sliding-plate viscometers: the sandwich viscometer, the sliding-plate viscometer, and the plate-and-tray viscometer for mobile liquids.

With the sandwich viscometer, a band of Mylar is sandwiched between two fixed plates and the fluid to be tested is placed in the spaces between the band and the plates, (Figure 16). For a given clearance between the band and the plates, and for a given force applied to the band, the drawing speed of the band through the test fluid is a measure of the fluid viscosity. A commercially available band viscometer has been developed by the National Printing Ink Research Institute of Lehigh University.

With the sliding-plate viscometer, the fluid sample is placed between two vertical parallel plates one of which is fixed. A force is then applied to the moveable plate and the plate movement is measured as a function of time. Knowing the thickness of the sample film, the drawing force applied, and the plate dimensions, it is then possible to calculate the shear stress, shear rate, and viscosity.

FIGURE 16 BAND VISCOMETER



The Shell-Hallikainen Sliding-Plate Microviscometer, a commercially available instrument, can be used to determine the flow properties of any fluid that will adhere to the sample plates to form a film. This instrument can also be used to study time-dependent properties of a fluid by observing the relationships of the shear stress with time.

For fluids that will not adhere to the plates of a sliding-plate viscometer, the plate-and-tray arrangement can be used. With this method a thin film of test fluid is placed in a shallow dish and a horizontal force is applied to a plate resting on the fluid film. From the instrument dimensions and the motion of the plate as a function of time, it is then possible to calculate the fluid viscosity.

DETERMINATION OF THIXOTROPIC BEHAVIOR

Many materials exhibit a time-dependent flow behavior in addition to the more common non-Newtonian behavior. As discussed earlier, these fluids are classified as being either rheopectic or thixotropic. Rheopectic behavior is rare, whereas, thixotropic behavior is observed in paint, ketchup and other foods; in some polymeric solutions, and in some oils. Thixotropic behavior, therefore, is of much greater practical importance than rheopectic behavior.

All of the viscometers discussed are suitable for measuring the flow properties of Newtonian fluids, since their flow properties are uniquely defined by just a single point on the flow curve. When a non-Newtonian fluid, however, is considered the entire flow curve is required to describe the flow properties. This means that only those instruments that are designed to measure the shear stress-shear rate relationship at a sufficient number of different shear rates (or shear stresses) to fully describe the flow curve, can be used. For instance, with a rotational viscometer, the flow curve may be obtained by measuring the torque produced at various rotational speeds of the cup or bob.

To describe the flow properties of a thixotropic fluid,

the entire flow curve must be determined for various durations of shear. Capillary viscometers are not well suited for quantitatively studying thixotropy, although they can be used to indicate, by the method already discussed, the presence of thixotropy. Rotational viscometers, on the other hand, are ideally suited for studying time-dependent effects. This is because the same sample of fluid remains in the instrument for any prescribed period of time.

There are at least two approaches to the quantitative determination of thixotropy. These are: (1) the measurement, by the method of Green (R-25), of the hysteresis between the flow curve plotted for the shear rate increasing and the curve plotted for the shear rate decreasing and (2) the determination of the decay of shear stress as a function of time at one or more constant shearing rates. The first method may be regarded as the classical approach, although the second method was investigated concurrently.

Green suggests that the criterion of thixotropy should be the hysteresis loop on the shear stress-shear rate curve. If a material is thixotropic, the flow curves obtained for the shear rate increasing and for the shear rate decreasing at a constant rate will not coincide but will form a loop. Green (R-25) has worked out methods for analyzing the shape and size of the hysteresis loop to obtain a quantitative

measure of the degree of thixotropy. The application of these hysteresis loops to equipment design has not been demonstrated, therefore, they are of academic interest only.

The measurement of the shear stress decay, on the other-hand, is a powerful tool which can be readily applied to equipment design. The flow behavior of a thixotropic fluid is fully defined by an experimental plot of shear stress versus time for various shear rates. The complete logarithmic shear stress-shear rate diagram for the thixotropic fluid may also be obtained simply by replotting the experimental data, providing data is obtained at a minimum of three shear rates.

APPLICATION OF RHEOLOGICAL DATA TO PIPELINE DESIGN

Any of the instruments already discussed will yield rheological data which may be applied to pipeline design providing the fluid under study is Newtonian. In fact, the design calculations for Newtonian fluids are well understood and well known by the pipeline engineer. The difficulty arises both in measuring and applying the rheological data when the fluid considered shows marked non-Newtonian behavior, and particularly when the fluid behavior is time-dependent.

For a Newtonian fluid, the pressure gradient in a horizontal pipe is given by the well-known Fanning equation:

$$\frac{\Delta P}{L} = \frac{2 f V^2 \rho}{g_c D} \qquad (17)$$

where $\frac{\Delta P}{L}$ = pressure gradient, lb_f per ft^2/ ft pipe

f = friction factor, dimensionless

V = average velocity, ft. per sec.

ρ = density lb_m per ft^3

D = inside diameter of pipe, ft

g_c = dimensional conversion factor $lb_m ft/ lb_f \ sec^2$

The friction factor depends upon whether the fluid motion is laminar or turbulent, as indicated by the Newtonian Reynolds number:

$$R_e = \frac{DV\rho}{\mu} \qquad (18)$$

where μ = the fluid viscosity $lb_m/ft \ sec$

In laminar motion, $Re < 2,100$, the friction factor is given by the relationship:

$$f = \frac{16}{Re} \qquad (19)$$

It can be seen that combining equations (17) and (19) yields the familiar Hagen-Poiseuille equation, equation (4) discussed earlier.

In turbulent flow, $Re >$ about 3,000, the friction factor is a function of the relative roughness of the pipe as well as the Reynolds number. For such cases the friction factor

is usually obtained from a friction factor-Reynolds number-relative roughness chart.

For time-independent non-Newtonian fluids, pipeline design is complicated by the fact that the ratio of the shear stress to the shear rate is not a constant. This means that, the consistency of the fluid will vary across the diameter of the pipe due to variations in the shear rate perpendicular to the direction of flow, and that the Newtonian Reynolds number, equation (18), will not apply.

Metzner (R-37) and Wilkinson (R-64) discuss the derivation of a number of relationships for calculating the pressure drop-flow rate relationship assuming various rheological models. Their approach, which can be used if the fluid flow behavior is represented by the power law relationship of equation (3), involves the use of the Fanning equation with special definitions of the friction factor and the Reynolds number. The modified Reynolds number is given by:

$$R_{e\,mod} = \frac{D^n V^{2-n} \rho}{\gamma} \qquad (20)$$

where n is a flow behavior index approximated by the slope on logarithmic co-ordinates of the shear stress-shear rate relationship; and

$$\gamma = K' \tau^{n-1} \qquad (21)$$

$$K' = K \left(\frac{3n+1}{4n} \right)^n \qquad (22)$$

where $\frac{K}{g_c}$ is the intercept on the shear stress axis at unity rate of shear of the tangent to the flow curve, on logarithmic co-ordinates, at the rate of shear of interest.

For the special case of Newtonian fluids, $n = 1.0$ and $K = \mu$, equation (20) reduces to:

$$Re = \frac{DV\rho}{\mu} \tag{18}$$

Combining equations (20), (21) and (22) yields:

$$Re_{mod} = \frac{D^n V^{2-n} \rho}{K} \cdot \frac{8}{\left(6 + \frac{2}{n}\right)^n} \tag{23}$$

Experimental evidence suggests that if Re_{mod} is less than about 3,000, the flow is laminar, otherwise it is turbulent. In the laminar flow region, the friction factor, by definition, is given by:

$$f = \frac{16}{Re_{mod}} \tag{24}$$

In the turbulent region the friction factor is a complex function of Re_{mod}, n and the pipe relative roughness. The friction-factor-Reynolds number correlation has not been well established for the turbulent region, however, in most cases it is possible to estimate a friction factor from the work of Dodge and Metzner (R-16) and Shaver and Merrill (R-52).

For laminar flow of a power law fluid, the rate of shear of the fluid at the wall of the pipe is given by (R-16).

$$\frac{dV}{dX_{wall}} = \left(\frac{1+3n}{4n}\right) \frac{8V}{D} \tag{25}$$

where $\frac{V}{D}$ is the average shear rate.

This relationship may easily be solved for the shear rate, $\frac{dV}{dx}$ if the fluid obeys the power law relationship given by equation (3). However, if the fluid does not obey the power law, then equation (25) must be solved for the shear rate, $\frac{dV}{dx}$, and the flow behavior index, n, using trial and error methods in conjunction with the consistency curve. Once the shear rate and corresponding flow behavior index have been found, the modified Reynolds number and hence the pressure gradients in the laminar flow region may be determined.

For turbulent flow, Dodge and Metzner (R-16) recommend that the flow properties be evaluated at a unit shearing stress equal to that which will exist at the wall. This is given by the fundamental relationship (R-23):

$$\frac{dF}{dA_{wall}} = \frac{D\Delta P}{4L} \qquad (26)$$

Trial and error calculations are again required. A wall shear stress, $\frac{dF}{dA}_{wall}$, is assumed and the corresponding flow properties are obtained from the consistency curve. From the modified Reynolds number and an appropriate friction factor chart, the pressure gradient is calculated using the Fanning equation. The assumed wall shear stress is checked using equation (26).

If the fluid considered displays an apparent yield value, a "start up" pressure gradient is required to initiate flow. This is calculated from equation (26) with the wall

shear stress taken as the yield stress.

When pipelines for time-dependent fluids are designed, consideration must be given to the fact that the fluid consistency not only varies across the diameter of the pipe, but that it will also vary, with time, in the direction of flow until an equilibrium state has been achieved. Govier and Ritter (R-23) have suggested a procedure for calculating the pressure gradients for time-dependent fluids.

Their method requires a knowledge of the shear stress-shear rate curves for the fluid at various durations of shear as well as the apparent yield value before flow will start. With this data, which may most conveniently be obtained using a rotational viscometer, the "start up" pressure gradient, the pressure gradient at any time, and the ultimate pressure gradient corresponding to an infinite duration of shear, may be calculated by the methods already discussed for a fluid which obeys the power law relationship.

PROPOSED VISCOMETER FOR TIME-DEPENDENT FLUIDS

Ritter (R-46) has shown that a number of the crude oils found in Alberta exhibit unusual flow properties, particularly thixotropy, at the temperatures encountered during storage and pipeline transmission. It would be useful, therefore, to

have an instrument which would measure the rheological properties of such oils, and present the data in a manner directly applicable to pipeline design. Since the properties of these fluids change with handling, or storage, it would be desirable to actually measure the fluid properties in the same environment as the fluids are encountered without transporting a sample to a laboratory. At present no such industrial instrument exists. With this in mind, a viscometer for studying time-dependent fluids is proposed which would be suitable for field installation.

As envisioned the instrument would consist of four cups rotating at different speeds around four bobs suspended by torsion wires. Provided the torsion wires are not deformed beyond their elastic limit, the shear stress at the bob wall would be calculated for each unit from the angular deflection of the bob by the equation (R-46):

$$\tau = a\theta \tag{27}$$

where τ = the shear stress

θ = the angular deflection

a = a proportionality constant.

The constant "a" is a function of the properties and diameter of the wire and the geometry of the system. It can be evaluated by calibration with the viscometer using a Newtonian fluid of known viscosity. The deflection of the bob pro-

duced by rotation of the cup at several predetermined speeds is noted, and the instrument constant "a" evaluated by comparison of equations (1) and (27).

For Newtonian fluids, the shear rate at the bob wall would be determined for each cup, from the cup speed and the geometry of the viscometer, using the following relationship established by Reiner and Riwlin (R-45a):

$$-\left(\frac{dV}{dR}\right)_{R_b} = \frac{4\pi S R_c^2}{R_c^2 - R_b^2} \qquad (28)$$

where S = rotational speed of the cup RPS

R_b = radius of the bob, ft

R_c = radius of the cup, ft.

Equation (28) will be useful when the instrument is calibrated. Normally, however, the shear rate at the bob wall would be determined using the Krieger and Maron (R-32) equation, for non-Newtonian fluids, in the following modified form (R-46):

$$-\left(\frac{dV}{dR}\right)_{R_b} = \frac{4\pi S R_c^2}{R_c^2 - R_b^2}\left[1 + B_1\left(\frac{1}{m} - 1\right) + B_2\left(\frac{1}{m} - 1\right)^2\right] \qquad (29)$$

where m = the slope, at any desired speed, of the logarithmic curve representing shear stress as a function of cup speed.

B_1 and B_2 = constants relating to the geometry of the instrument and defined by:

$$B_1 = \frac{R_c^2 - R_b^2}{2R_c^2}\left[1 + \frac{2}{3}\ln\frac{R_c}{R_b}\right] \qquad (30)$$

$$B_2 = \frac{R_c^2 - R_b^2}{6R_c^2}\ln\frac{R_c}{R_b} \qquad (31)$$

A study of the various viscometers discussed in the literature has revealed a number of methods for determining the angular deflection. The two most common methods are (1) visual and (2) electrical detection of the angular deflection. In the proposed instrument the torsion wires will be completely submerged in the test fluid. Any attempt to transmit the angular motion of the bob to the outside of the instrument case by means of a rigid rod is precluded by the high frictional damping of any bearing or seal. The transmission of a light beam through the test fluid for detection by a photo-electric cell or even the visual observation of the bob position are also precluded by the expected opaqueness of many sample fluids. Visual means of detecting the deflection angle are thus impractical with the proposed instrument.

Electrical means of detecting the deflection angle have the best possibilities of success since they can be made essentially frictionless and highly accurate. The systems used in the Rotovisco, the Agfa and the Polard rotation viscometers were considered, but found to be unsatisfactory for this application. The potentiometer system

in the Rotovisco was eliminated because it was not frictionless and hence would cause some difficulty in determining a yield value. The linear differential transformer system in the Agfa was eliminated because the detector heads would have to be submerged in the test fluid and hence the angular motion would be severely damped. The variable capacitance method of the Polard was eliminated because of the large condenser plate area required to give the instrument a sufficiently wide range.

The method for detecting the angular deflection of the bob, which was finally decided upon, was developed by Associate Professor Y. J. Kingma and Research Assistant J. A. Ash of the Department of Electrical Engineering. Their method, to be discussed in a later section, involves a completely frictionless device and relies upon the determination of the time between a pulse generated by the bob and a reference pulse.

Power for the rotating outer cups would be derived from a synchronous motor which rotates the cups through a series of gears. A two-speed clutch arrangement would be located between the motor and the main gear train. Ideally the cup speeds should be arranged so that each successive speed is a constant whole number ratio of the preceding faster speed. Since the desired speed range of the rotating cups

is 600 RPM to 10 RPM, the proposed speeds are:

1st cup = 640 RPM

2nd cup = 160 RPM

3rd cup = 40 RPM

4th cup = 10 RPM

With the two-speed clutch arrangement, it would be possible to further reduce all the cup speeds by one-half.

The gear train driving the rotating cups would be inside the case of the viscometer, but would be isolated from the test fluid by a gear train housing. Mechanical seals would be used to prevent the test fluid entering the housing around the cup drive shafts. The torsion wires would pass coaxially through the cup drive shafts.

To allow fresh sample to enter the annular spaces between the bob and cup assemblies, the cups would be designed so that the top and bottom could be opened. As proposed, the bottom of the cup would be lowered and the cup wall would be allowed to fall under its own weight until stopped by drive pins passing through the cup lid (see Figure 19). Test fluid would then be free to circulate between the cup wall and the bob.

A means of manually rotating the bob would also be provided. This feature would permit resetting of the de-

flection angle detection system in the event that the test fluid shows a substantial yield value. It would also permit the detection of the initial yield value by a method to be discussed later.

The four rotating cups and associated gear train housing would be mounted vertically in a section of steel pipe. Although the instrument would be operated at essentially atmospheric pressure, the system should be designed to withstand approximately 200 psi which might be expected while filling the viscometer from a pipeline. The necessary valves and overpressure safety devices would also be provided to conform with the oil field safety standards.

PROTOTYPE VISCOMETER DESIGN

To check the mechanical design details of the proposed viscometer and to evaluate the method for detecting the angular deflection of the bob, a prototype viscometer was built. The prototype design has all the features of the proposed viscometer except that only one rather than four cup and bob assemblies are provided, and a six inch diameter lucite pipe rather than a steel pipe is used as the instrument case.

Figures 17, 18 and 19 show photographs of the prototype viscometer and associated equipment. While Figures 20 and 21 show the plan and front elevation of the prototype viscometer, Figures 22 to 26 show various sectional views. Detail drawings are shown in Appendix I.

The heart of the viscometer is the hollow lucite bob and rotating lucite cup. The bob has a diameter of 1.520 inches and an overall length of 3.0 inches and is provided with conical ends having an apex angle of 150 degrees. The rotating cup has an inside diameter of 1.700 inches and is provided with a removable top and bottom which may be opened to allow a fresh fluid sample to enter the annular space between the cup and bob. Both the top and bottom of the cup are tapered in an effort to reduce the cup end-effects.

FIGURE 17 PROTOTYPE VISCOMETER

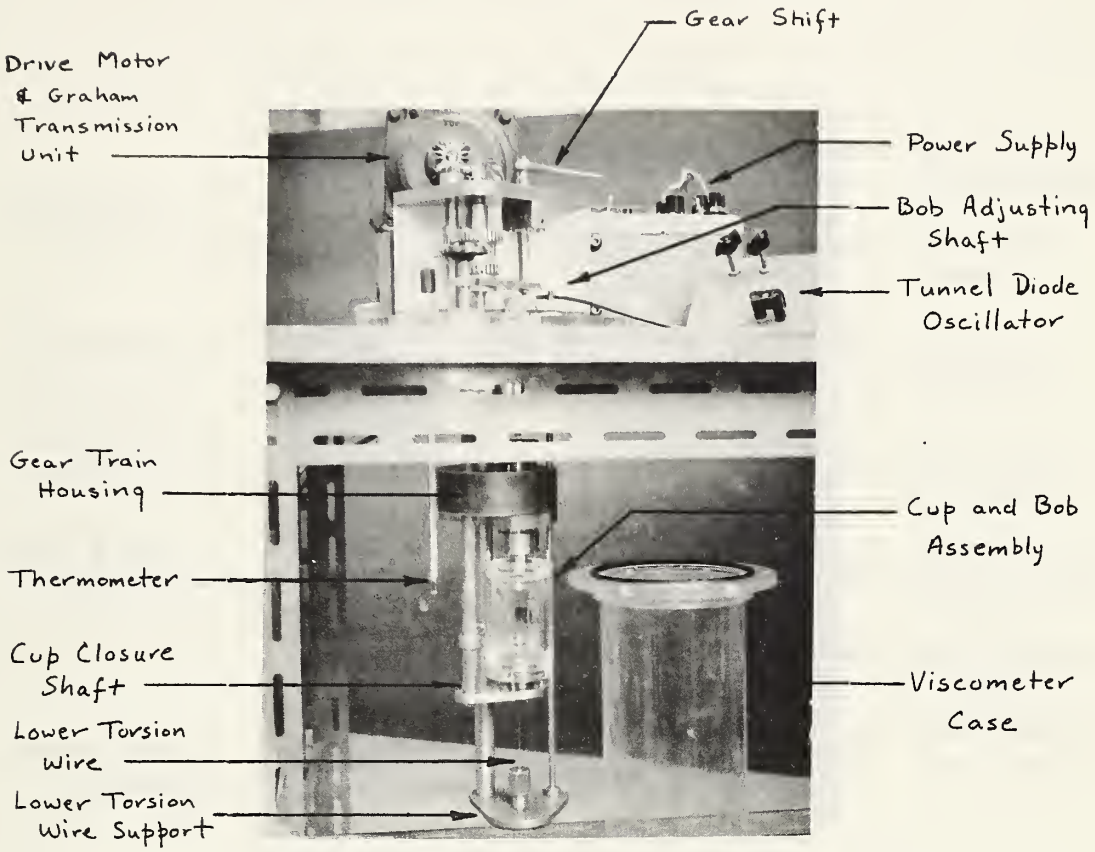


FIGURE 18 PROTOTYPE VISCOMETER - LOWER SECTION

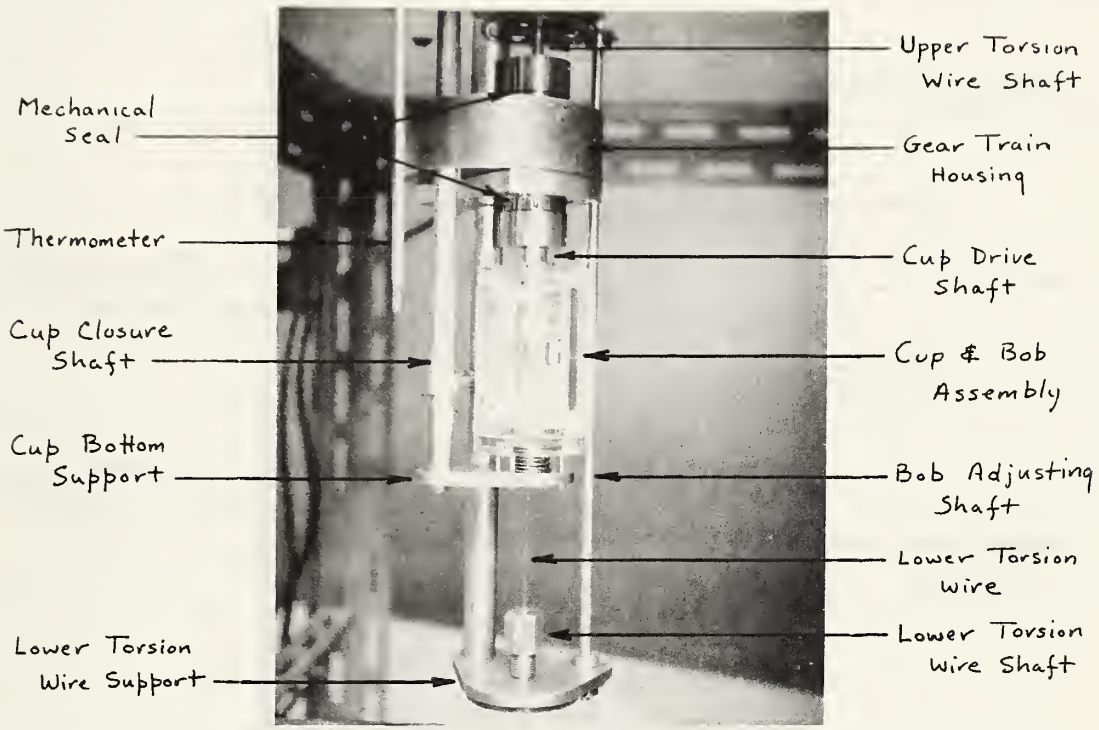


FIGURE 19 PROTOTYPE VISCOMETER - CUP & BOB

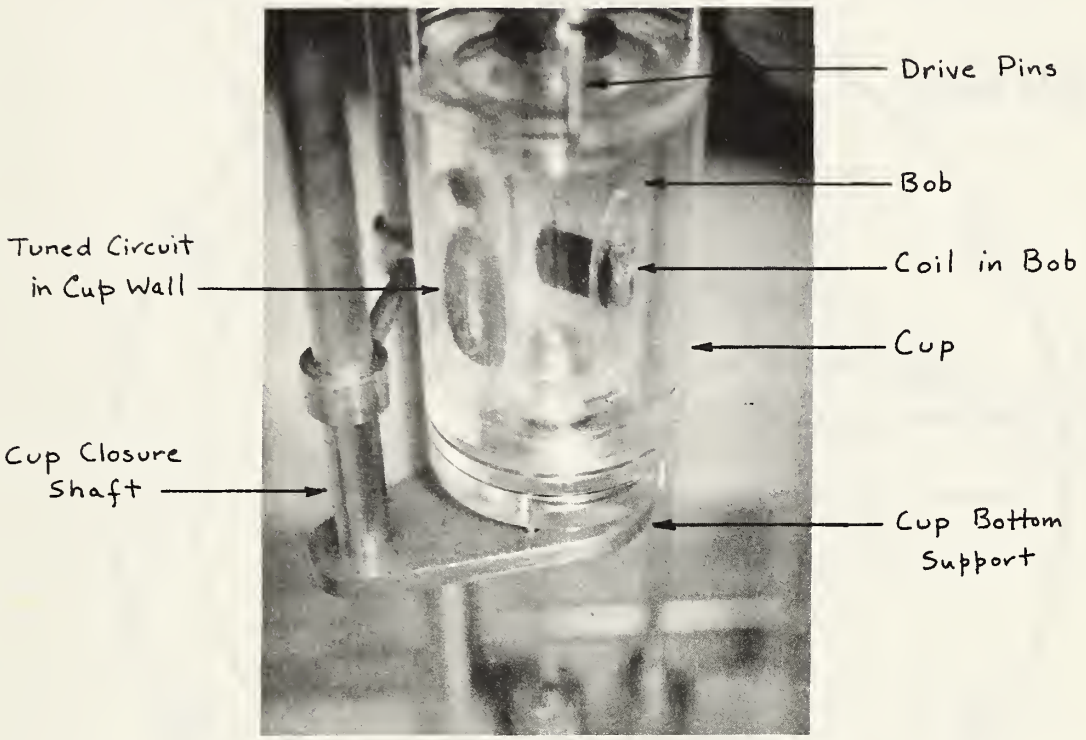


FIGURE 20 PROTOTYPE VISCOMETER - PLAN

(Scale $\frac{1}{2}$" = 1")

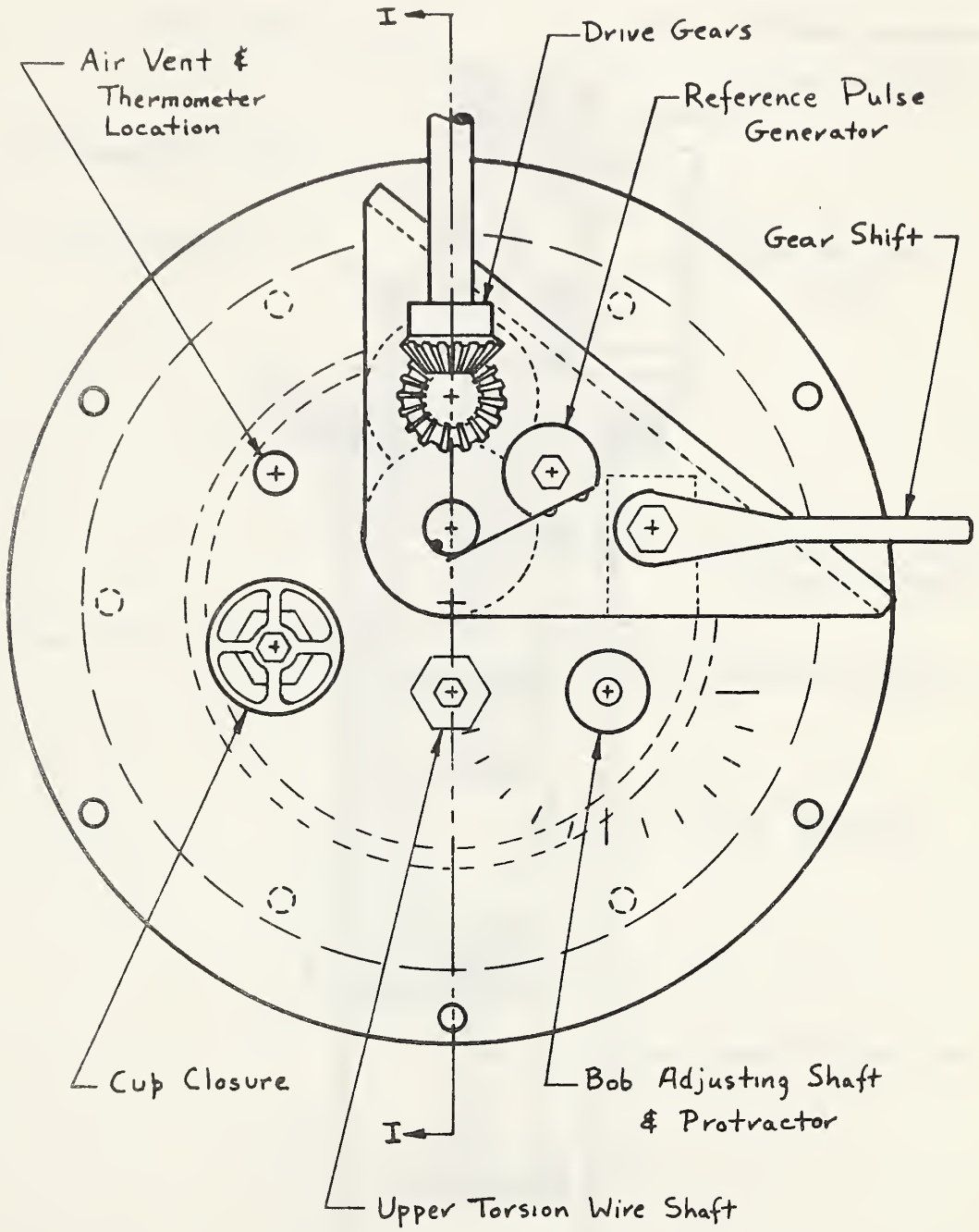


FIGURE 21 PROTOTYPE VISCOMETER - FRONT ELEVATION

(Scale $\frac{1}{4}" = 1"$)

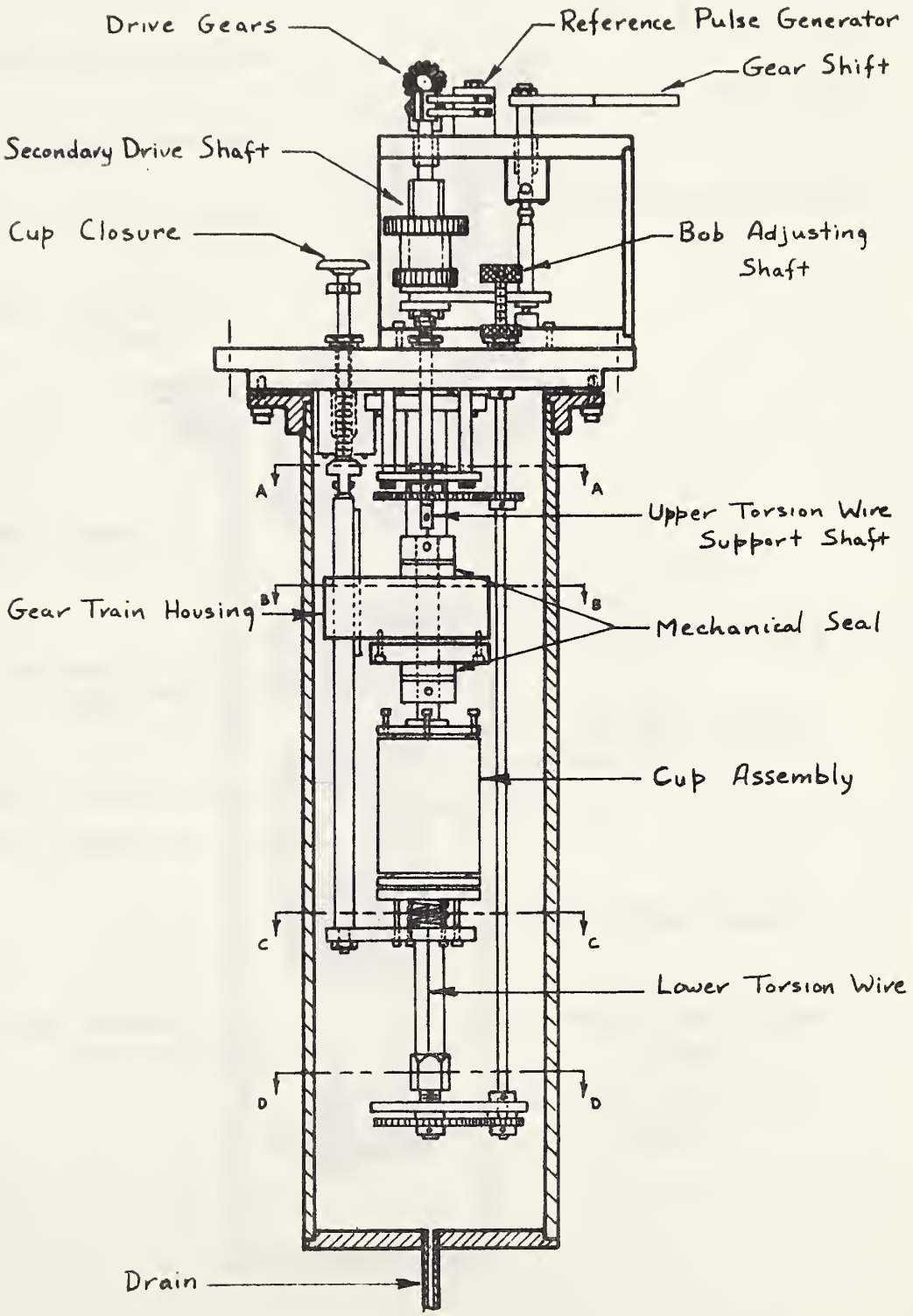


FIGURE 22 PROTOTYPE VISCOMETER – SECTION I-I

(Scale $\frac{1}{4}" = 1"$)

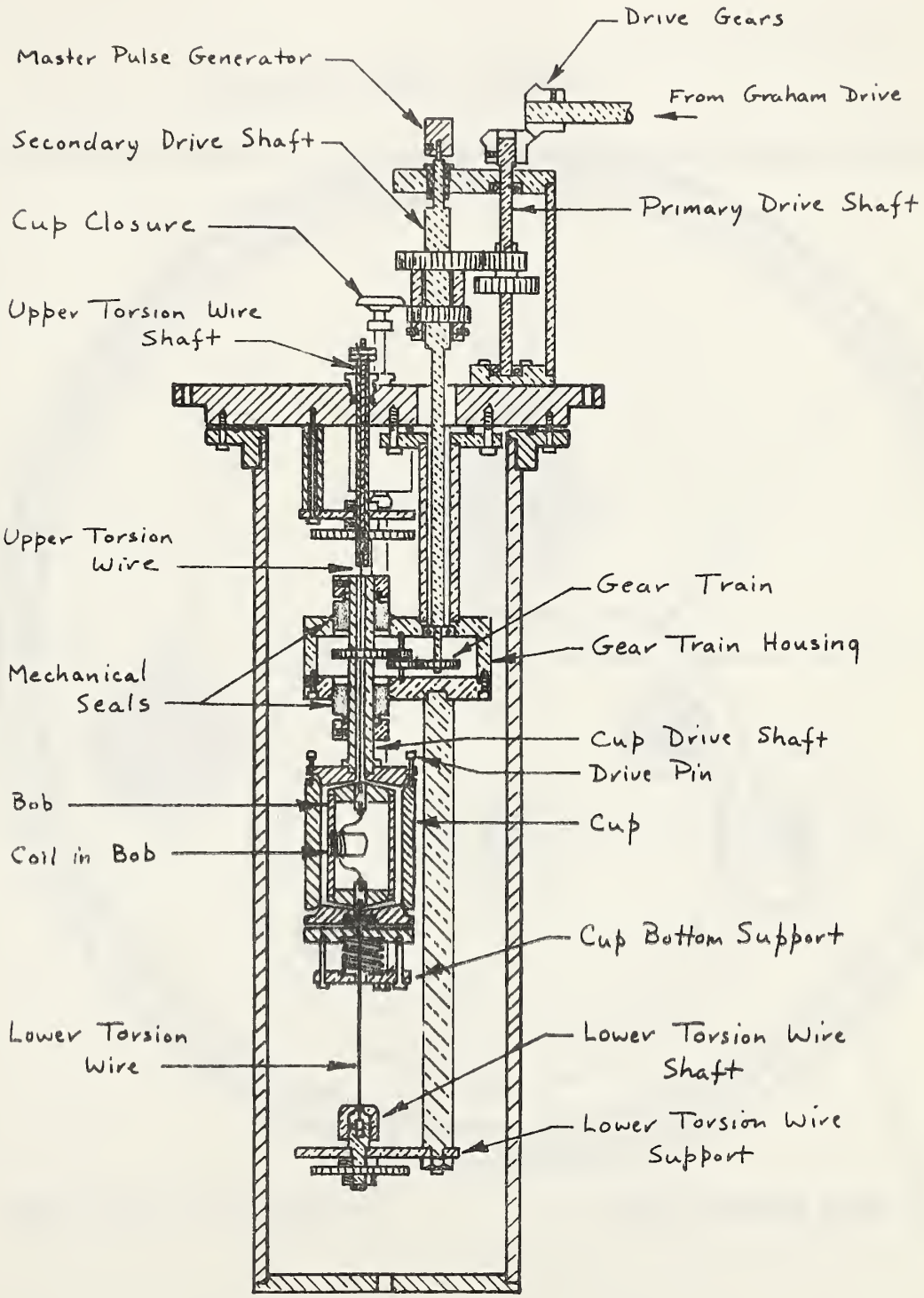


FIGURE 23 PROTOTYPE VISCOMETER – SECTION A-A

(Scale: Full Size)

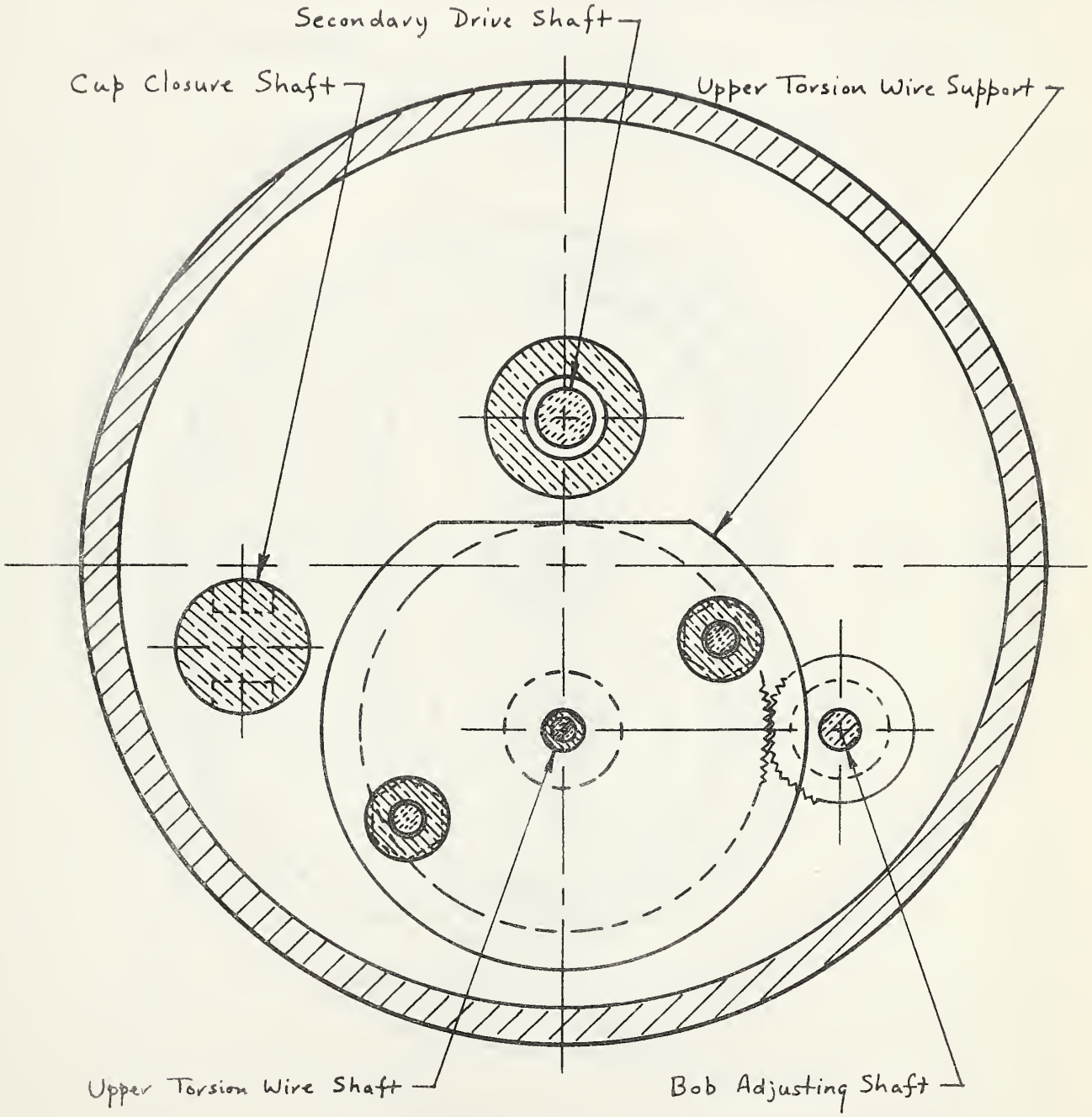


FIGURE 24 PROTOTYPE VISCOMETER - SECTION B-B

(Scale: Full Size)

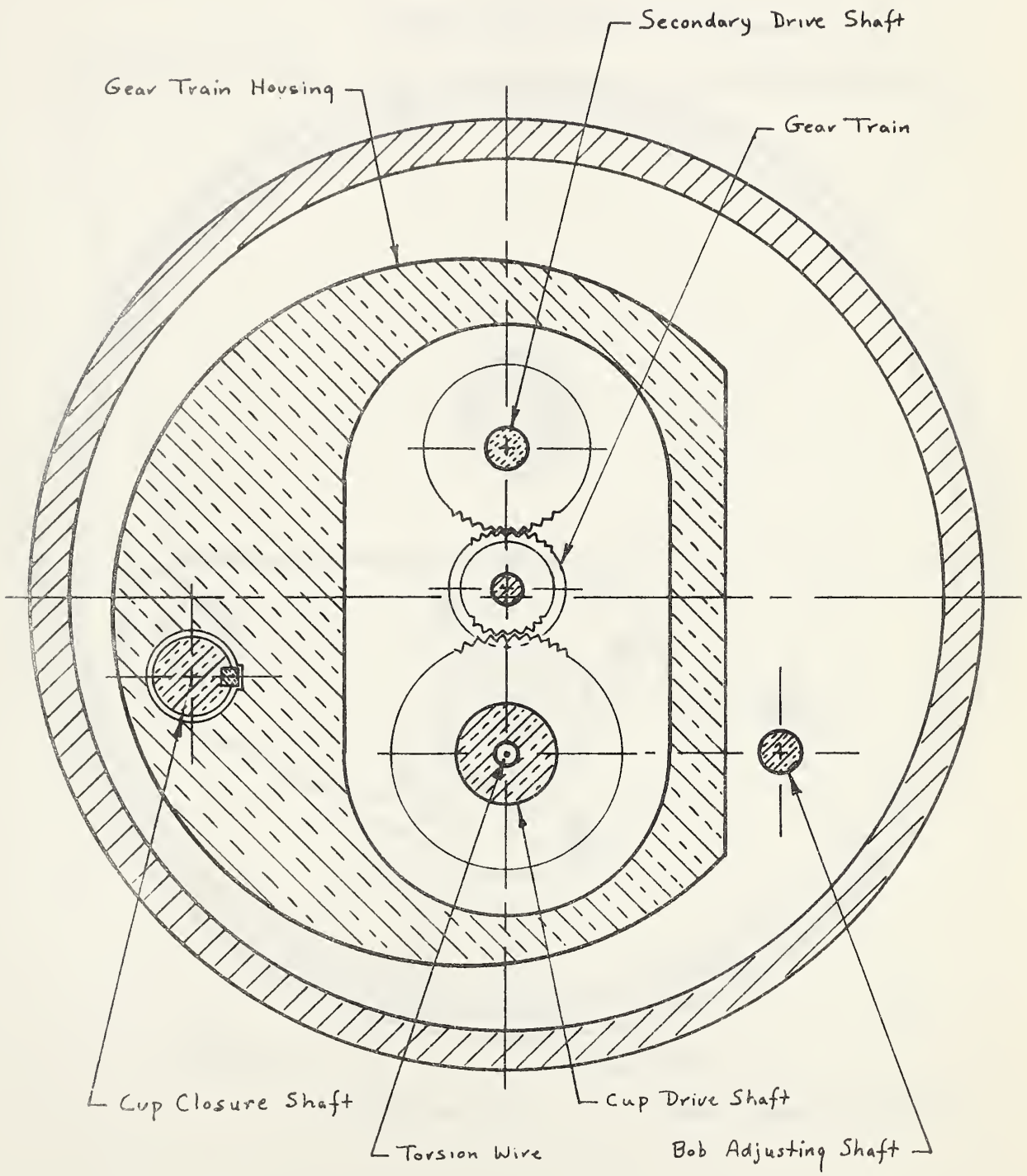


FIGURE 25 PROTOTYPE VISCOMETER - SECTION C-C

(Scale: Full Size)

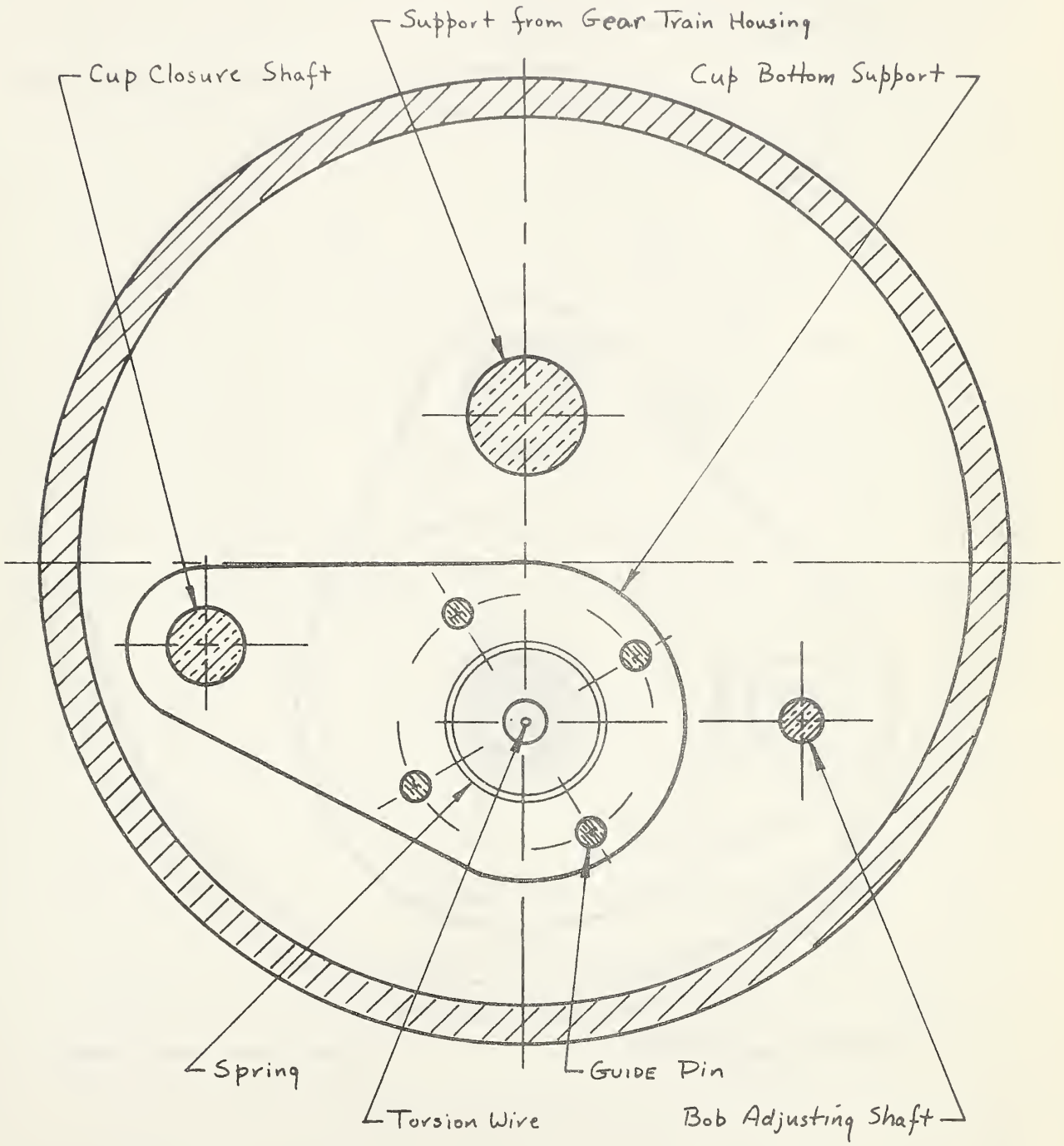
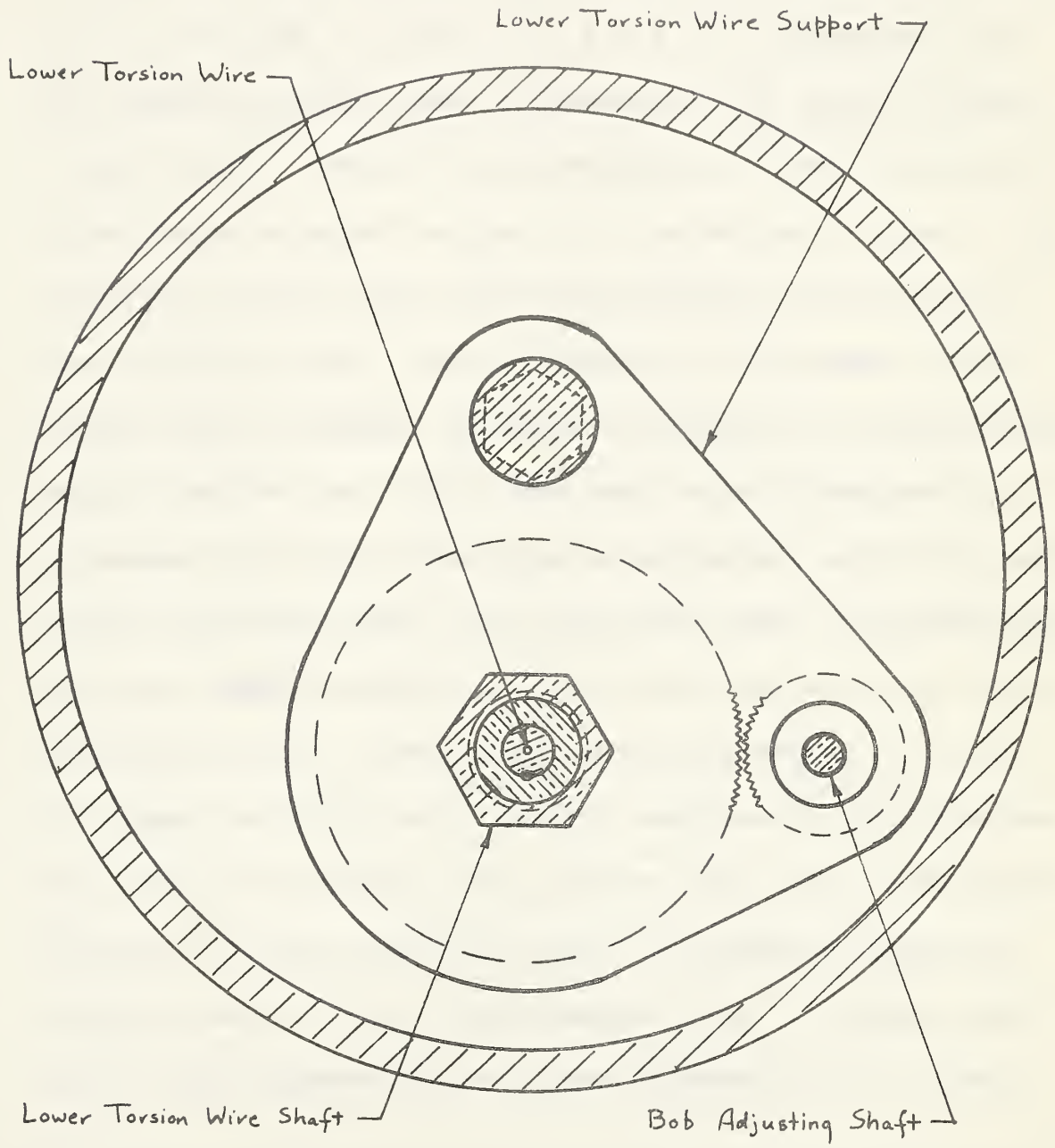


FIGURE 26 PROTOTYPE VISCOMETER - SECTION D-D

(Scale: Full Size)



When the cup is closed, the tapered ends of the cup and the conical ends of the bob form a common apex as shown in Figure 22.

The cup is driven at any desired speed within the range of 0 to 570 RPM, by means of a 1/4 H. P. synchronous motor and Graham Variable Speed Transmission unit acting through a gear train. The gear train consists of four 32 Diametral Pitch gears arranged to give a 2:1 reduction in speed. The diameters of the gears, from the secondary drive shaft to the cup drive shaft, are 1.0 inches, 0.750 inches, 0.562 inches and 1.5 inches, as shown in Figure 22. To protect the gears from the test fluid, the gear train is enclosed in a housing, and John Crane Type 9A mechanical seals are used on the cup drive shaft. The cup drive shaft is pressed into the top of the cup which in turn drives the cup wall through four drive pins. A gear shift providing either a 1:1 or a 2:1 speed reduction and a neutral position is placed between the Graham Transmission unit and the gear train. The speed selection is made while the motor is stopped by engaging either a pair of 1 1/2 inch diameter 20 D. P. change gears or a 2 inch diameter and a 1 inch diameter 20 D. P. gear.

The cup is opened and closed by the action of a shaft operating on the principle of a rising stem valve. To open the cup, the plate supporting the bottom of the cup is lowered

thus allowing the cup wall to fall under its own weight. The travel of the cup wall is limited by the shoulders on the drive pins which make contact with the cup lid. The travel of the cup bottom support plate is limited by a stop placed on the cup closure stem. Since the total travel of the cup bottom support is one inch and the travel of the cup wall is one half inch, the cup is opened at both top and bottom.

When the cup is closed, both the top and bottom must seal tightly on the cup wall, otherwise test fluids which exhibit the Weissenberg effect will tend to pump through the annular space between the cup and bob. For this reason, the cup top and bottom fits into the cup wall, as shown in Figure 22 and Figures I-1, I-2 and I-3, thus ensuring a good seal.

The cup bottom is constructed of two pieces of lucite separated by a ball bearing. The upper portion is free to rotate with the cup wall, while the lower portion is prevented from rotating by four guide pins located in the cup bottom support plate. A spring located between the cup bottom support plate and the cup bottom is provided to prevent excessive pressure being exerted on the cup by the cup closure.

The bob, Figure I-4, is held within the rotating cup by

two five inch long calibrated torsion wires. For this model a 24 B & S gauge MacMichael Viscometer wire was used. The upper torsion wire passes coaxially through the cup drive shaft and is attached to an insulated shaft which extends through the top of the instrument casing. The lower torsion wire is attached to a shaft held in the lower torsion wire support which in turn is suspended from the gear train housing. The torsion wires are kept under a constant tension by means of a spring and compression nut forming part of the lower torsion wire shaft (Figure I-6).

To manually rotate the bob, for purposes to be discussed later, two 2.5 inch diameter, 64 D. P. gears are provided on the two shafts holding the upper and lower torsion wires. These gears are engaged by two 0.938 inch diameter gears located on the bob adjusting shaft which extends through the top of the instrument case. When required, a pointer may be attached to the knurled knob on the top of the bob adjusting shaft to indicate the amount of rotation on a protractor. Since the gear ratio is 8:3 a $2^{\circ}40'$ rotation of the bob adjusting shaft yields a one degree rotation of the bob.

A master pulse and a pulse generated by the bob and cup arrangement are utilized to measure the angular deflection of the bob. Initially both the master pulse generator and the cup and bob are arranged so that the master pulse and the

pulse from the bob are on the point of firing. Once the cup starts to rotate, the bob will be deflected by the viscous drag of the sample fluid and there will be a time lag between pulses. From this time lag and the speed of the cup, the angular deflection of the bob may be calculated.

The pulse from the bob is generated by means of a circuit developed by Associate Professor Y. J. Kingma and Research Assistant J. A. Ash, of the Department of Electrical Engineering The block diagram of the circuit is shown in Figure 27a. The basis of the system is a Bistable Tunnel Diode Oscillator which has the ability to oscillate at two amplitude levels. A simplified diagram of the Bistable Tunnel Diode Oscillator is shown in Figure 27b. Initially the two variable resistances A and B (Figure 27b) are adjusted to permit stable oscillation at the lower limit cycle. Variable resistance A is used to adjust the bias on the Tunnel Diode, while variable resistance B is used to adjust both the bias on the Tunnel Diode and the quality of the tuned circuit. The coil of the tuned circuit is wound on a piece of 1/2 x 1/8 x 3/4 inch Ferrite and is located in the center of the hollow lucite bob with the face of the coil as close to the outside surface of the bob as possible.

Another tuned circuit is imbedded in the wall of the cup as close to the inside surface as possible. As this tuned

FIGURE 27 ELECTRICAL CIRCUITS - BLOCK DIAGRAMS

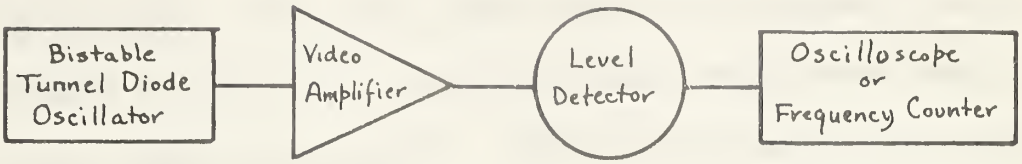


FIGURE 27a TUNNEL DIODE PULSE GENERATOR

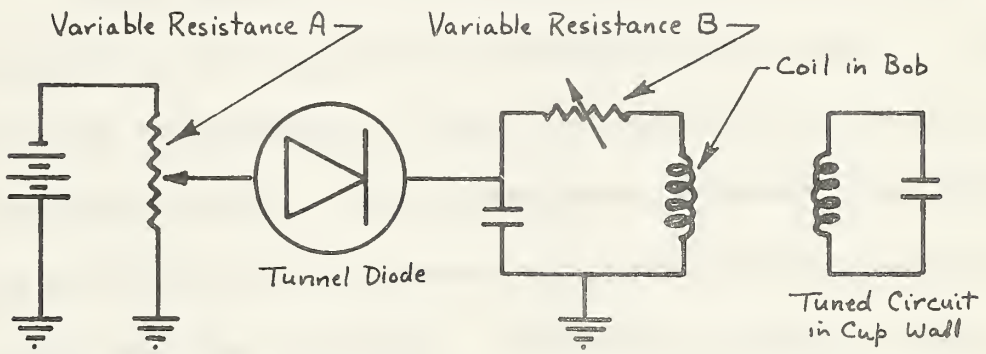


FIGURE 27b BISTABLE TUNNEL DIODE OSCILLATOR

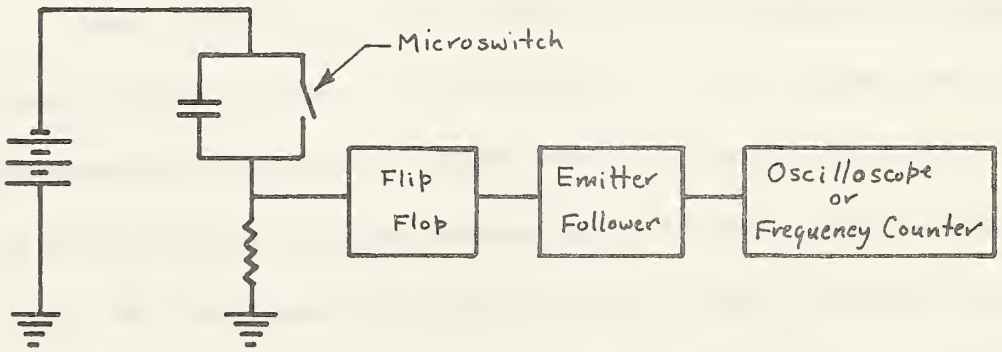


FIGURE 27c MASTER PULSE GENERATOR

circuit passes the coil mounted in the bob, an A. C. Resistance is reflected into the Tunnel Diode Oscillator circuit such that the Tunnel Diode Oscillator switches to the upper limit cycle. Once the tuned circuit has passed the coil mounted in the bob, the Tunnel Diode Oscillator again reverts to the lower limit cycle. After amplification and level detection a 10 volt pulse is produced. Figure 19 shows the coils mounted in the cup and bob assembly.

The master pulse is generated by a pulse generator located on the top of the secondary drive shaft. Initially a brush arrangement as shown in Figure 18 was used to generate the master pulse. The arrangement, however, was found unsatisfactory and a standard microswitch was finally used to calibrate the instrument. The block diagram of the master pulse system is shown in Figure 27c.

The location of the master pulse generator causes some complications. Since there is a 2:1 speed reduction between the secondary shaft and the cup drive shaft, there are two master pulses generated for every revolution of the cup. To overcome this difficulty a flip flop was introduced into the circuit of the cup. On every pulse, the flip flop changes the polarity of the signal to the oscilloscope or frequency counter. Since both the oscilloscope and the frequency counter can be set to trigger for either a positive

or negative change in polarity, one pulse can essentially be eliminated. An emitter-follower was added to the circuit in an effort to eliminate false triggering due to local disturbances such as motor noise and mechanical vibrations.

The time lag between pulses may be calculated from the positions of the master pulse and the cup pulse on the oscilloscope or may be measured directly by a frequency counter set to read the period time. In the case of the oscilloscope, the master pulse is used to trigger the oscilloscope and the position of the cup pulse is observed. The time lag can then be calculated if the sweep time of the oscilloscope beam is known. In the case of the frequency counter, the master pulse triggers the counter while the cup pulse stops it. This gives the time between pulses as a digital display.

PROTOTYPE VISCOMETER CALIBRATION

To evaluate the mechanical and electrical design features of the proposed viscometer, three Newtonian fluids of known viscosity were tested. Since there is no temperature control on the prototype viscometer, the viscosities of the test fluids had to be determined after the calibration runs at the average temperature encountered during the test. Kinematic viscosities were determined using a Cannon-Fenske viscometer according to ASTM Standard D445-53T, and the fluid densities were determined using a Christian-Becker Chainomatic Gravitometer. The fluids used were various mixtures of Primol D (a white mineral oil) and Varsol and had the following fluid properties.

TABLE 1 TEST FLUID VISCOSITIES

Fluid	Temp ^{o}F	Kinematic Viscosity Centistokes	Density gm/cc	Absolute Viscosity	
				Centipoise	lb /sec ft.
A	79	122.31	0.8732	106.80	0.0717
B	82	93.22	0.8705	81.15	0.0546
C	84	68.59	0.8677	59.51	0.0401

To calibrate the viscometer, approximately two gallons of the test fluid was introduced into the instrument case by means of a tygon hose attached to the drain at the bottom of the instrument. The cup was then closed, and the relationship

between cup speed and the angular deflection of the bob determined.

The time lag between a master pulse and the pulse generated by the cup passing the bob, was used to determine the angular deflection of the bob from the relationship.

$$\theta = \frac{\Delta t}{t_c} \times 360^\circ \qquad (32)$$

where θ = the deflection angle in degrees

Δt = the time difference between a master pulse and the pulse from the bob

t_c = the time for one revolution of the cup.

The application of equation (32) requires that both the master pulse and the pulse from the bob be set to occur simultaneously when the cup is not rotating. To assure this, the instrument is zeroed, before testing a fluid, by rotating the secondary drive shaft until the master pulse is just firing, and then rotating the bob by means of the bob adjusting shaft until the pulse from the bob is also just firing. Extreme care must be taken to ensure that both the bob and the secondary shaft and hence the cup, are rotated in the same direction as during normal operation. In the prototype design, this means that both the secondary shaft and the bob adjusting shaft must be rotated in a counter-clockwise direction.

The speed of the cup was checked using a frequency counter,

an oscilloscope, a Strobotac strobe light, and a Sanborn recorder. All methods were fairly dependable although the frequency counter was considered most accurate. For this reason, it was used to measure the period of one revolution of the cup, and was also used, as described earlier, to determine the time lag between pulses.

The data obtained from the calibration runs are shown in Table 2. The deflection angles are calculated from equation (32) and the speed in RPS is taken as the reciprocal of the period time in seconds.

Since the fluids used for calibrating the instrument are Newtonian, the shear rate at the bob wall may be determined from the Reiner and Riwlin equation, equation (28). Also since the viscosities of the test fluids are known, the shear stress in the fluid may be found from equation (1). From the bob deflection angle and equation (27) the instrument constant, a, may then be calculated (Appendix II). The results of these calculations for the three test fluids are shown in Table 3.

The instrument constant, a, may also be obtained from the slope of the deflection angle versus the rotational speed curve for a Newtonian fluid of known viscosity. By combining equations (1), (27) and (28), it may be shown (Appendix II) that the instrument constant for the prototype viscometer is

TABLE 2 CALIBRATION DATA

For Fluid A

Run	Cup Average Period millisec/rev	RPS	Time Between Pulses millisec	Angle Degree	Temp. °F.
1	1371.0	0.728	57 (52-62)	14.95	77
2	447.8	2.23	54	43.5	
3	276.7	3.61	52.7	68.3	
4	211.1	4.74	52.1	88.8	
5	170.8	5.85	51.9	109.2	
6	139.1	7.19	51.8	134.0	
7	111.7	8.95	51.7	167.0	81
8	3640.0 (3636-3645)	0.275	48.0	4.75	

For Fluid B

1	318.9	3.14	35.0	39.4	81
2	210.6	4.75	35.0 (33-38)	59.6	
3	685.4	1.46	35.5 (33-37)	18.65	
4	965.5	1.04	35.0	13.05	81.5
5	1396.5	0.717	32.0 (28-34)	8.25	

For Fluid C

1	421.8	2.37	24.1	20.6	
2	663.0	1.51	24.3	13.2	
3	1156.0	0.855	22.0	6.85	
4	305.4	3.28	24.5	28.9	
5	236.9	4.22	25.2	38.3	
6	211.4	4.72	25.4	43.3	
Gear Change					
7	1638 (1624-1662)	0.611	32.2 (31.5-32.8)	7.08	
8	551.0	1.815	24.4	15.9	84.5
9	197.5	5.06	25.4	46.3	
10	137.4	7.27	25.7	67.3	
11	105.5	9.48	25.5	87.0	85.0

TABLE 3 CALCULATION OF INSTRUMENT CONSTANT

For Fluid A

Run	RPS	Angle Degree	Shear rate from (28) sec^{-1}	Shear stress from (1) lb_f/ft^2	Instrument Constant from (27) lb_f/ft^2 degree
1	0.728	14.95	45.3	0.101	0.00675
2	2.23	43.5	138.8	0.310	0.00712
3	3.61	68.3	224.7	0.501	0.00735
4	4.74	88.8	295.1	0.685	0.00741
5	5.85	109.2	364.2	0.812	0.00744
6	7.19	134.0	447.6	0.998	0.00745
7	8.95	167.0	557.1	1.242	0.00744
8	0.275	4.75	17.1	0.038	0.00800

For Fluid B

1	3.14	39.4	195.5	0.332	0.00843
2	4.75	59.6	295.7	0.503	0.00844
3	1.41	18.65	87.8	0.149	0.00799
4	1.05	13.05	65.4	0.111	0.00851
5	0.717	8.25	44.6	0.076	0.00921

For Fluid C

1	2.37	20.6	147.5	0.184	0.00893
2	1.51	13.2	94.0	0.117	0.00886
3	0.855	6.85	53.2	0.066	0.00964
4	3.28	28.9	204.2	0.255	0.00882
5	4.22	38.3	262.7	0.328	0.00856
6	4.72	43.3	293.8	0.367	0.00848
7	0.611	7.08	38.0	0.047	0.00664
8	1.815	15.9	113.0	0.141	0.00887
9	5.06	46.3	315.0	0.393	0.00849
10	7.27	67.3	452.6	0.565	0.00840
11	9.48	87.0	590.1	0.736	0.00846

given by:

$$a = \frac{\mu (62.25)}{g_c \, q}$$

where q = the slope of the curve relating the deflection angle and the rotational speed for the appropriate fluid.

The deflection angle-rotational speed curves for the three test fluids are shown in Figure 28. From these curves the instrument constant was calculated as shown in Appendix II.

The constants obtained both graphically and by calculation for each test fluid are tabulated below.

TABLE 4 INSTRUMENT CONSTANT

Test Fluid	Instrument Constant $lb_f/ft^2 degree$	
	Graphical	Calculated Average
A	0.00746	0.00737
B	0.00828	0.00852
C	0.00827	0.00856

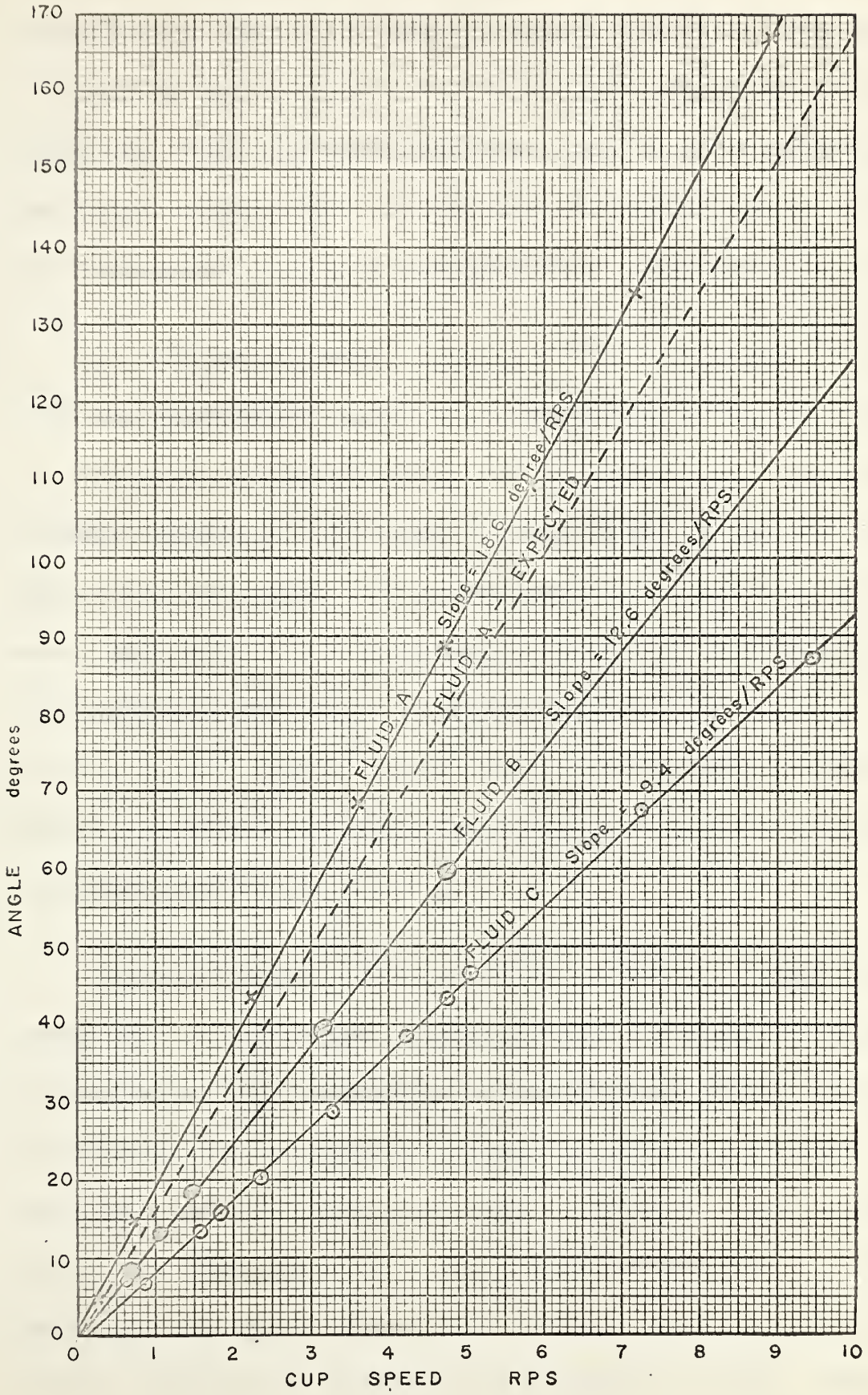
The constants obtained using test fluids B and C show reasonably good agreement, although there is considerable scatter as seen in Table 3. Most of the scatter is undoubtedly caused by a wobble in the motion of the cup which causes the bob to oscillate. This oscillation, in turn, causes the pulse from the bob to occur slightly early or late depending on the position of the bob.

The large discrepancy between the constant obtained from Fluid A and the constant obtained from the other test fluids is inexplicable. As shown in Figure 28, the actual curve has a larger slope than the curve using an instrument constant of 0.00828 lb_f/ft^2 degree. The rise in temperature and resulting decrease in viscosity that occurred during the testing does not account for the discrepancy between Fluid A and the other fluids. In fact, the rise in temperature would show just the opposite trend. The possibility of an error in determining the viscosity of the test fluid was investigated. However, a redetermination of the viscosity showed that the viscosities used were correct.

There are two possible causes of the anomalous behavior of Fluid A, neither of which, unfortunately, can be confirmed. Firstly, it is possible that the choice of the average temperature at which the fluid viscosity was determined was in error. If the viscosity at approximately 76°F. rather than at 79°F. was used, the data for Fluid A would lie very close to the expected curve shown in Figure 28. There is, however, no justification for using a temperature of 76°F.

Secondly, it is possible that the torsion wire had been damaged or was failing by fatigue. If this were the case, the instrument constant obtained from Fluid A would likely be closest to the actual value, and the higher constant

FIGURE 28 DEFLECTION ANGLE vs CUP SPEED



obtained from Fluids B and C would likely be due to work hardening of the wire. Shortly before commencing the testing of Fluid A, the torsion wires were overstressed and the day after the test runs were completed, the lower torsion wire was found broken. The reliability of the torsion wires and the instrument constants obtained must, therefore, be doubted.

An attempt was made to predict the instrument constant from the viscometer calibrations performed by Ritter (R-46). Not too much weight, however, can be placed on these calculations (Appendix III) since the magnitude of the bob end effect was not known, and an assumed value, based on Ritter's work, had to be used. However, even with this assumption, the predicted instrument constant was found to be $a = 81.6 \times 10^{-4}$ $lb_f/ft^2 degree$, which lies between the experimental instrument constants obtained from Fluid A and that obtained from Fluids B and C.

The experimental determination of the instrument constant for the prototype viscometer was a convenient means of evaluating the mechanical and electrical design features of the proposed viscometer. Even though the instrument constants obtained from the three calibration runs do not agree, the fact that the relationship between the deflection angle and cup speed (Figure 28) for all three fluids is linear, as would be expected for Newtonian fluids, indicates

that the mechanical design features of the new instrument are satisfactory. Furthermore, at no time could the discrepancy between the test runs be attributed to the viscometer design features. The prototype viscometer was never intended for the routine determination of fluid properties, but was built to evaluate the proposed viscometer design features. It is, therefore, not essential that a reliable instrument constant be determined at this time.

DISCUSSION OF OPERATION AND RECOMMENDATIONS

The mechanical operation of the prototype viscometer was generally good, although many design refinements are required. The method of detecting the angular deflection also worked satisfactorily, after some initial problems.

The major mechanical problem would seem to be alignment which, because of the high sensitivity of the angular detection system, is very important. With the present instrument design, it is very difficult to properly align the cup and bob assembly. To overcome this problem, it is proposed that guide rods be extended through the cup wall into the cup top and upper portion of the cup bottom. These rods would ensure alignment of the cup assembly since they would prevent the cup from sitting off vertical on the conical sealing surface of the cup bottom, and yet would not interfere with the opening or closing of the cup. It is also proposed that the cup assembly be dynamically balanced to eliminate any tendency to wobble. To align the torsion wires so that the bob is located exactly in the center of the cup, a jig or some method of optically aligning the wires will have to be provided. Once the wires are aligned, realignment should not be necessary, and the lower torsion wire support and the cup bottom support can

be pinned in place.

An electrical problem of false firing of the bob Tunnel Diode pulse generator is also probably due to mechanical misalignment. It was found that the angular detection system was so sensitive that even a slight oscillation of the bob, caused by poor alignment, could be detected. The distance between the coil mounted in the bob and the tuned circuit in the cup was also found to be very critical. Initially a bob with a diameter of 1.473 inches and a gap between coils of 0.224 inches was tried and found unsuitable. A new bob, however, with a gap between coils of 0.170 inches has worked satisfactorily.

For speeds below 1 RPS the viscometer and the angular detection system was quite unsatisfactory. At these low speeds, the cup period showed variations of approximately 1%, while the time between pulses showed a 10% variation as seen from Table 2. A comparison of Tables 3 and 4 shows that the instrument constant calculated at speeds below 1 RPS, deviate as much as 20% from the graphically determined instrument constant, while the deviation at speeds above 1 RPS is approximately 2%.

Some of the scatter of the data of Table 2 plotted on Figure 28, is undoubtedly due to mechanical misalignment and

resulting oscillation of the bob. Most of the scatter, however, is due to misfiring of the master pulse and false triggering of the frequency counter by electrical noise from the drive motor. Except at speeds of rotation below 1 RPS, most of the electrical noise feeding to the frequency counter has been eliminated by placing filters on all the power supply lines and on the leads to the frequency counter. The misfiring of the master pulse has not been solved. In fact, a satisfactory method of obtaining a clean square-wave master pulse has not been found. The system of brushes tried initially and shown in Figure 21 gave a very poor square-wave with many microsecond pulses superimposed on the basic square-wave. The microswitch arrangement finally used was satisfactory for short periods but the life of a microswitch in this application is too short for an industrial model. Although no proposal is made at this time, it might be necessary to generate the master pulse with a second Tunnel Diode oscillator, or a photoelectric cell.

The performance of the electrical circuitry would be further improved if a direct ground connection to the lower torsion wire support was provided. The present connection is through either the bob adjusting shaft or the cup closure shaft and in either case is unsatisfactory because of the poor electrical contact across the oil film between rotating components.

The importance of correctly zeroing the instrument can not be overemphasized. If the master pulse initially fires before the pulse from the bob, then the angular deflection obtained by equation (32) would be too large since the time between pulses, Δt, would be too large. Conversely, if the master pulse initially fires after the pulse from the bob, then the angular deflection obtained would be too small. The effect of the master pulse firing too soon is clearly demonstrated by Figure 29. In this case, the line passing through the origin is obtained by plotting the angular deflection-speed of rotation data for Fluid B given in Table 3. The other line (Table 5) shows the master pulse initially firing five degrees before the bob.

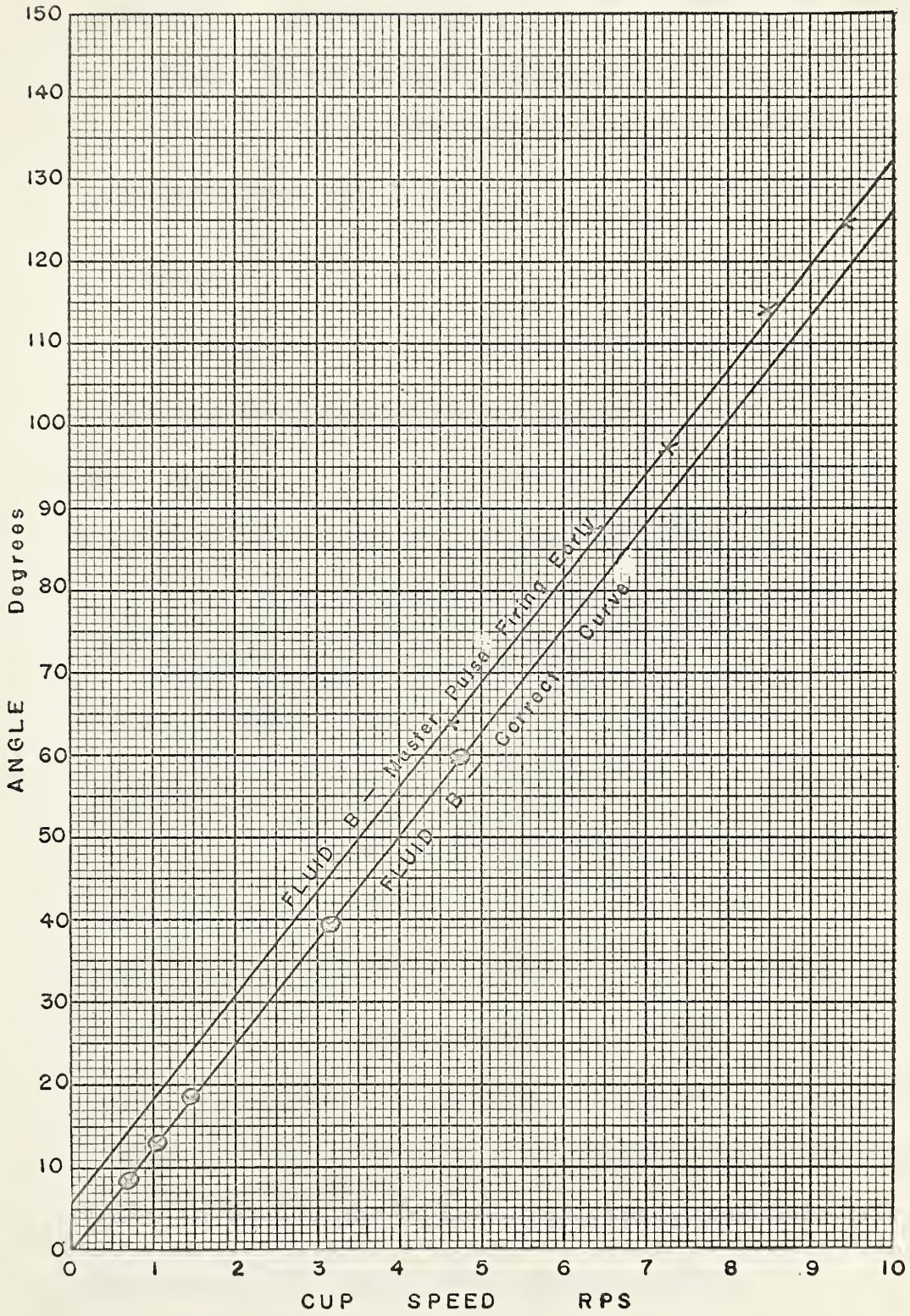
TABLE 5 EFFECT OF MASTER PULSE FIRING EARLY

For Fluid B (Absolute viscosity 81.15 centipoise).

Run	Cup Average Period millisec/rev	RPS	Time Between Pulses millisec	Angle degree
7	214.7	4.67	38.1	63.8
8	156.4	6.38	37.9	87.2
9	137.8	7.26	37.1	96.9
10	118.6	8.43	37.5	114.0
11	105.8	9.45	37.6	124.3

The principle of adjusting the bob via the torsion wires appears to be satisfactory although the operation of the bob adjusting shaft should be smoother. The use

FIGURE 29 EFFECT OF IMPROPER ZEROING OF THE VISCOMETER



of a larger gear ratio between the bob adjusting shaft and the torsion wire support shafts would be highly advantageous. Roller thrust bearing should also be provided to support the bob adjusting shafts. The possibility of replacing the o-ring seal, where the bob adjusting shaft comes through the instrument case, by a mechanical seal should be investigated.

If these modifications can be made to the bob adjusting shaft, it should be possible to accurately determine the yield stress of a fluid by the following procedure. With the instrument carefully zeroed by the method already discussed and the pointer attached to the knurled knob on the bob adjusting shaft, the torsion wire is rotated so that the bob tends to rotate. In this case, the bob must be rotated in the opposite direction to its normal rotation, that is, for the prototype viscometer the bob adjusting shaft would be rotated in a clockwise direction. The angle through which the torsion wires must be rotated in order that the master pulse and the pulse from the bob are no longer firing simultaneously is a measure of the shear stress at the bob wall that must be exceeded before the bob will rotate. The yield stress, therefore, can then be calculated from the yield angle using equation (27).

Another proposed mechanical design change involves the enlarging of the cup lid to provide a deeper threaded section for the cup drive shaft. It is felt that if the drive shaft is threaded further into the lid and keyed, better alignment of the drive shaft and cup lid could be achieved.

Cheaper mechanical seals should also be investigated. In the industrial model at least, eight, and possibly twelve (if mechanical seals are used on the bob adjusting shafts) mechanical seals are required. At the present price of $37.00 each, this item alone represents a substantial portion of the total cost of the proposed model.

FUTURE WORK

The ultimate aim of this work is to build and field test the proposed viscometer. From these field tests the applicability of the instrument for predicting pipeline pressure drops would then be evaluated.

Before the field model can be built, however, considerably more work must be done with the prototype model. The problem of aligning the cup and bob must be solved, and the method of zeroing the instrument must be made more reliable. It is also felt that the electrical system designed to detect the angular deflection of the bob is must too complicated for an industrial application. For this reason, the detection of an angular displacement by a variable capacitance will be investigated. The method was investigated briefly before the prototype viscometer was designed, but was discarded because of the low capacities that would be encountered. However, because of the simplicity of the method and direct functional relationship between angle and capacitance, the method should at least be tested and evaluated.

CONCLUSIONS

In this work, the various methods of determining the rheological properties of a fluid were reviewed, and some of the more common rheological instruments were discussed. The special methods of determining thixotropic behavior and the application of rheological data to the design of pipelines was also discussed.

A search of the literature revealed that there are no industrial instruments available which measure the rheological properties of a time-dependent fluid in such a manner that the data is directly applicable to pipeline design. For this reason, a new viscometer that could be installed in a field location and could be used for studying time-dependent properties of many industrial fluids, was proposed. The instrument as proposed consists of four cup and bob assemblies rotating at four different speeds. Each speed corresponds to a shear rate given by equation (29) and the angular deflection of the bob corresponds to a particular shear stress as given by equation (27). Thus, by measuring the angular deflection of each bob as a function of time, the rheological behavior of the test fluid may be represented by a shear stress decay curve (Figure 1d). The application of these curves to pipeline design was also discussed.

The design details and calibration of a prototype viscometer, built to test the novel features of the new instrument, were presented. A number of design weaknesses, which were observed during the calibration of the instrument, were discussed and some design improvements to be incorporated in the proposed viscometer were presented.

The electrical system for detecting the angular deflection of the bob was discussed briefly. The system, although it worked reasonably satisfactorily, was considered to be too complicated for an industrial application. For this reason, a simpler method, using a variable capacitance to determine the angular deflection, will be investigated.

In conclusion, it may be said that the prototype viscometer has demonstrated the feasibility of the proposed instrument. Therefore, although many design refinements are required, work will commence immediately on the design and construction of a field model of the proposed viscometer.

NOMENCLATURE

a	Radius of capillary, ft.
a	Instrument constant, lb_f/ft^2degree
B_1	Constant in Krieger and Maron Equation
B_2	Constant in Krieger and Maron Equation
D	Diameter of Capillary
f	Friction factor, dimensionless
G	Torque
g	Gravity constant ft/sec^2
g_c	Dimensional Conversion Factor, $lb_m ft/lb_f sec^2$
h	Depth of bob immersion, inches
K	Fluid consistency index $lb_m/ft\ sec^{2-n}$
k	Instrument constant, capillary viscometers
L	Length of capillary or tube, ft.
m	Slope on logarithmic curve representing shear stress as function of cup speed
n	Flow behavior index
ΔP	Pressure drop
γ	Slope of bob deflection vs cup speed curve
R	Radius
r	Radius of sphere
Re	Reynolds number
Re_{crit}	Reynolds number defined by stability criterion
Re_{mod}	Modified Reynolds number
S	Speed, RPS
t	Time, sec.
V	Velocity, ft/sec
v	Velocity, ft/sec
dv/dx	Shear rate, sec^{-1}

Greek Symbols

α	Angle between plate and cone
γ	Denominator of Re_{mod}
$\dot{\gamma}$	Shear rate, sec^{-1}
η	Coefficient of rigidity $lb_m/ft\ sec$
θ	Angular deflection
μ	Viscosity, $lb_m/ft\ sec$
ν	Kinematic Viscosity, ft^2/sec
ρ	Density, lb_m/ft^3
ρ_s	Density of falling sphere, lb_m/ft^3
τ	Shear stress, lb_f/ft^2
τ_y	Yield stress, lb_f/ft^2
Ω	Cup speed, radian/sec
ω	Angular velocity, radian/sec

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**References designated with a double asterisk were also unavailable and are quoted in Reference (R-29).

APPENDIX I

DETAIL DRAWINGS

Figure

I-1	Cup Lid
I-2	Cup Wall
I-3	Cup Bottom
I-4	Bob
I-5	Upper Torsion Wire Shaft
I-6	Lower Torsion Wire Shaft
I-7	Gear Housing
I-8	Gear Housing-Lower Portion
I-9	Gear Housing-Support

FIGURE I-1 CUP LID

Scale : Full Size

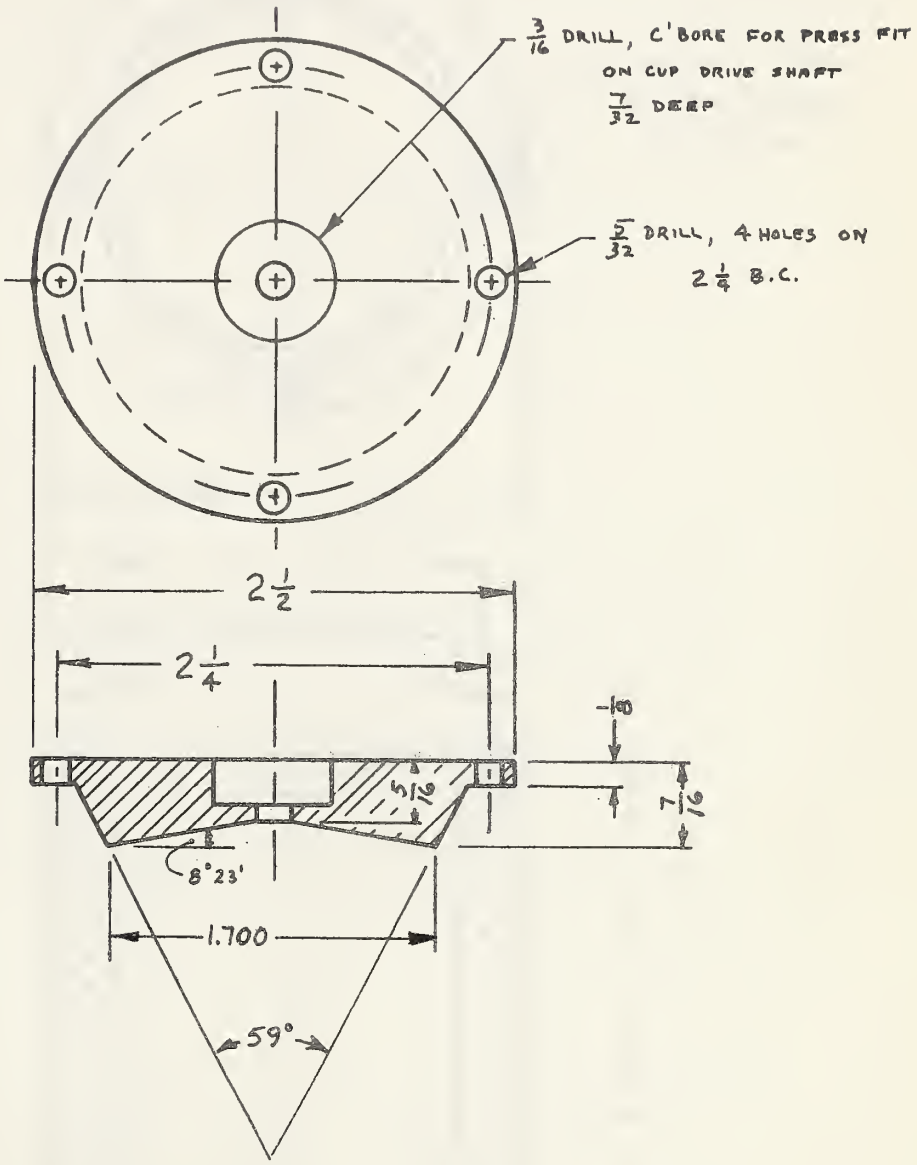


FIGURE I-2 CUP WALL

Scale : Full Size

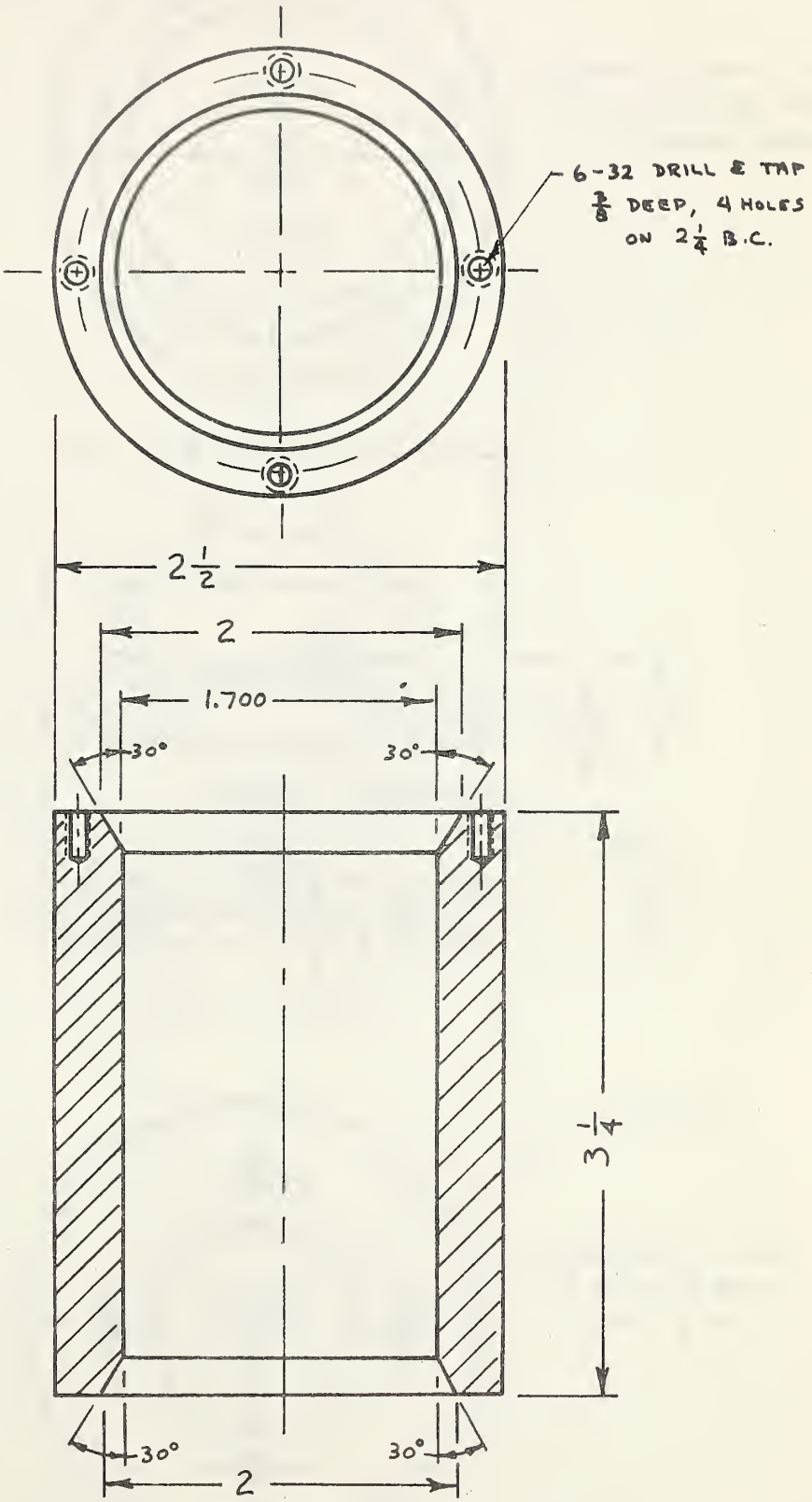


FIGURE I-3 CUP BOTTOM

Scale Full Size

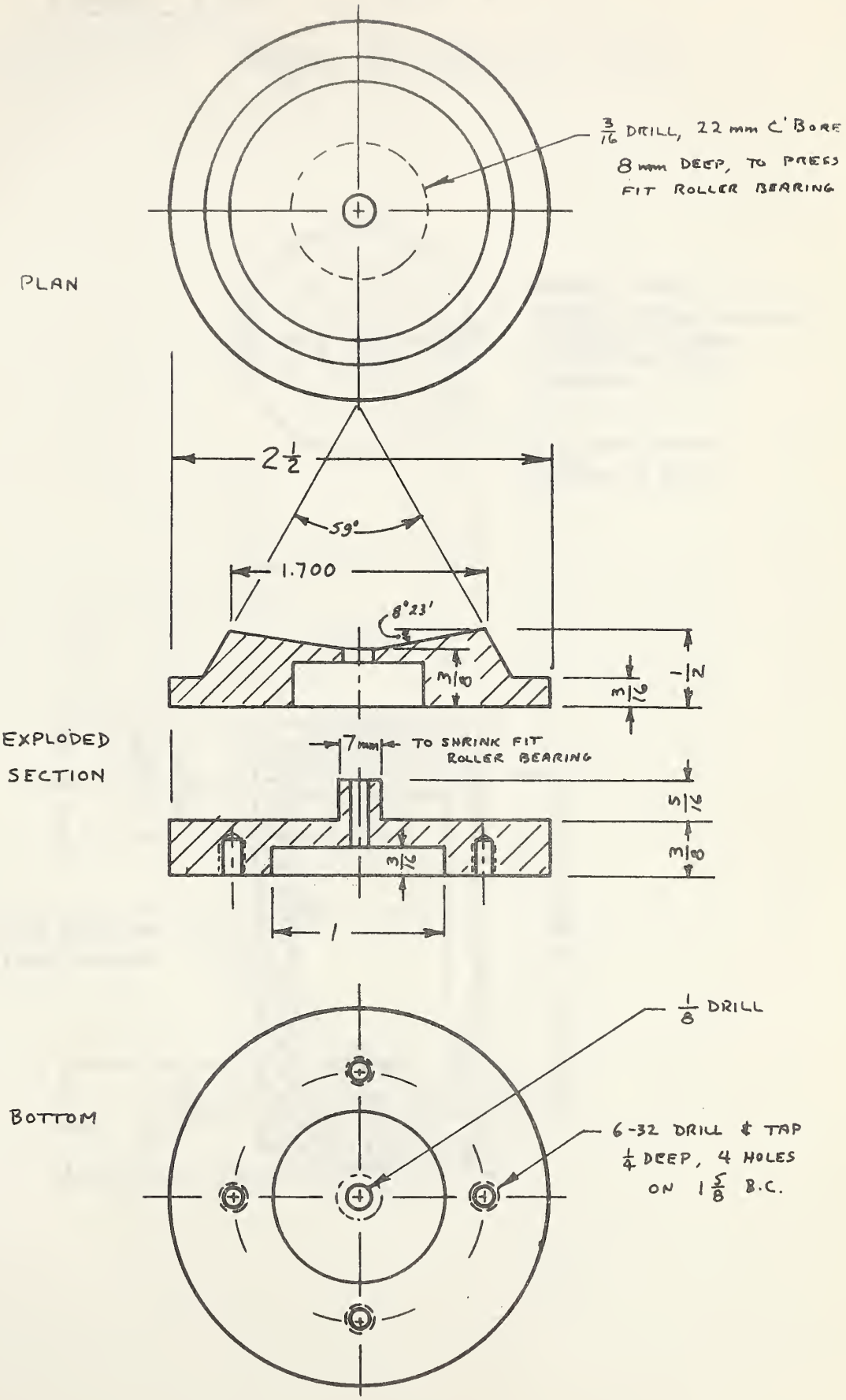


FIGURE I-4 BOB

Scale: Full Size

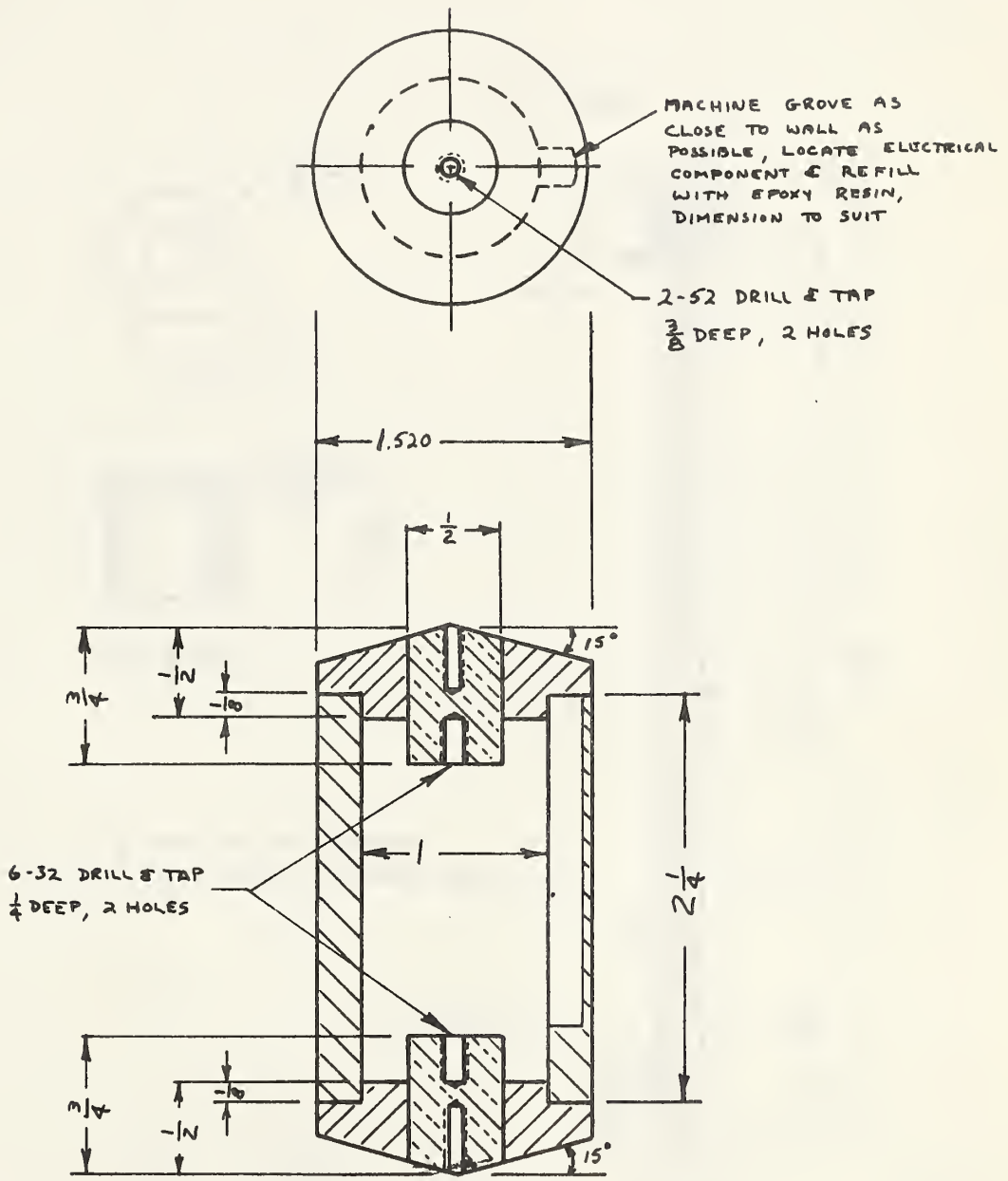

MACHINE GROVE AS CLOSE TO WALL AS POSSIBLE, LOCATE ELECTRICAL COMPONENT & REFILL WITH EPOXY RESIN, DIMENSION TO SUIT
2-52 DRILL & TAP
3/8 DEEP, 2 HOLES
1.520
1/2
15°
6-32 DRILL & TAP
1/4 DEEP, 2 HOLES
1
15°

FIGURE I-5 UPPER TORSION WIRE SHAFT

Scale: Full Size

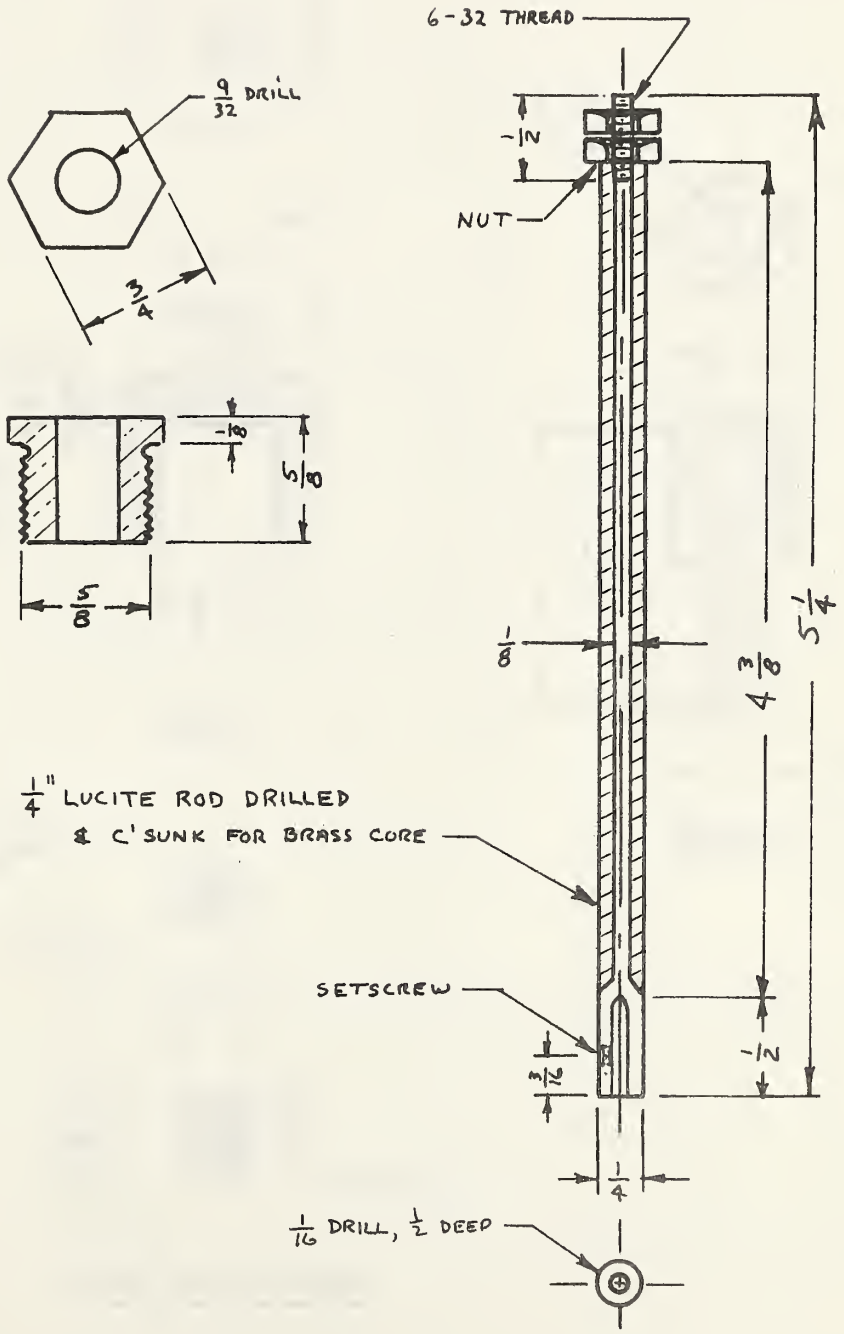
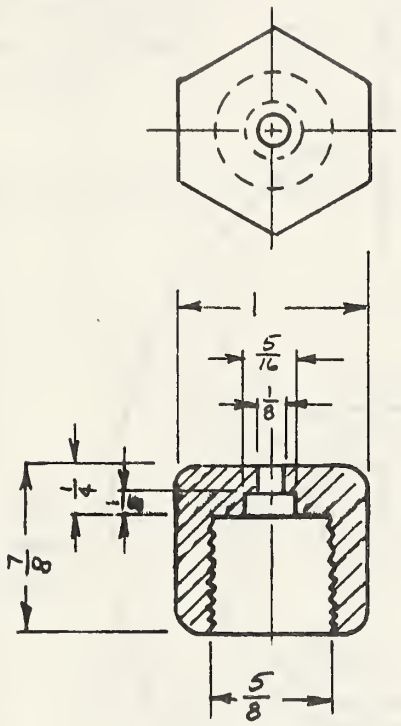
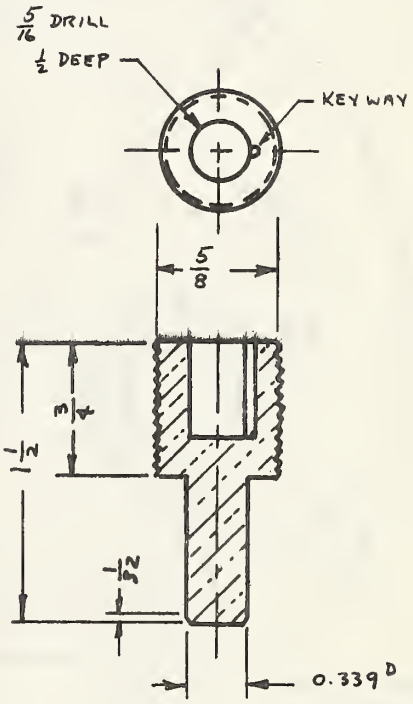


FIGURE I-6 LOWER TORSION WIRE SHAFT

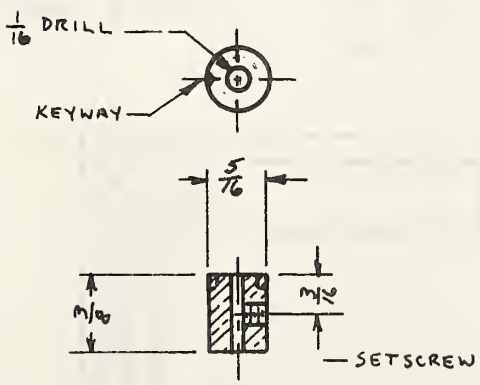
Scale: Full Size



NUT



SHAFT



WIRE END-PIECE

FIGURE I-7 GEAR HOUSING

Scale: Full Size

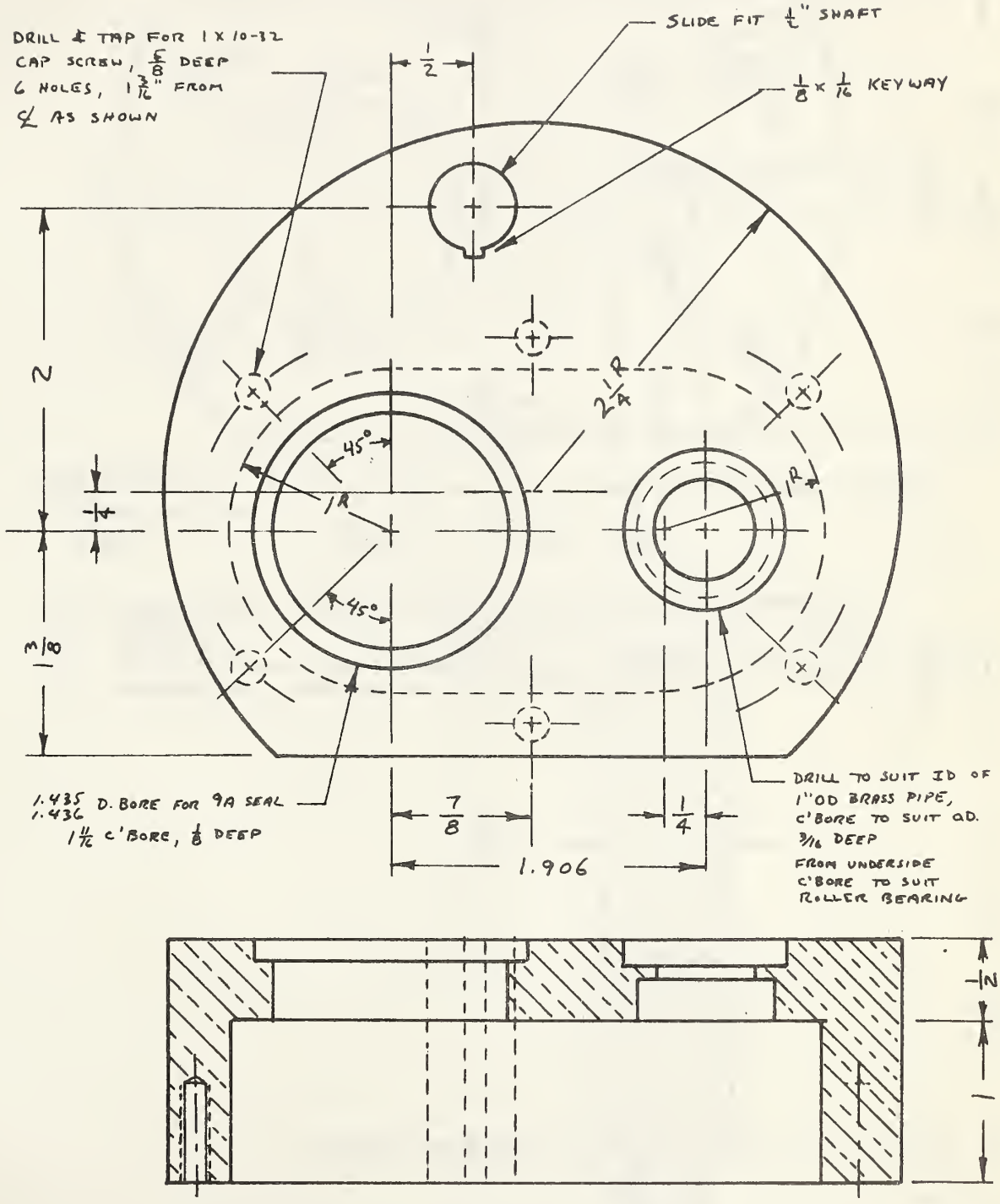

DRILL & TAP FOR 1 X 10-32 CAP SCREW, 5/8 DEEP 6 HOLES, 1 3/16" FROM ℄ AS SHOWN
SLIDE FIT 1/2" SHAFT
1/8 x 1/16 KEYWAY
1/2
N
M/00
1/4
45°
1R
2 1/4 R
1.435 1.436 D. BORE FOR 9A SEAL
1 11/16 C'BORE, 1/8 DEEP
7/8
1/4
1.906
DRILL TO SUIT ID OF 1" OD BRASS PIPE, C'BORE TO SUIT OD. 3/16 DEEP FROM UNDERSIDE C'BORE TO SUIT ROLLER BEARING

FIGURE I-8 GEAR HOUSING - LOWER PORTION

Scale: Full Size

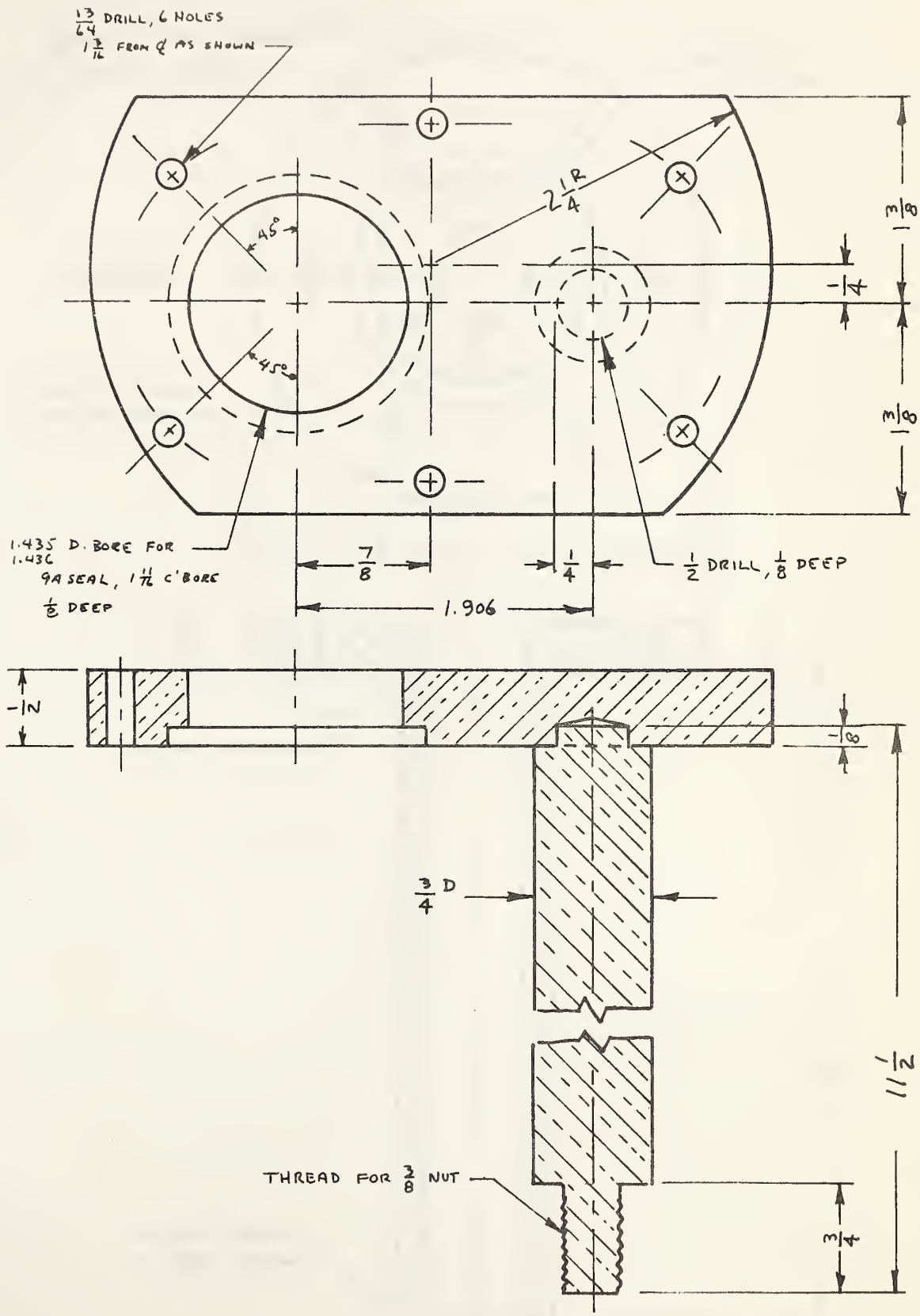
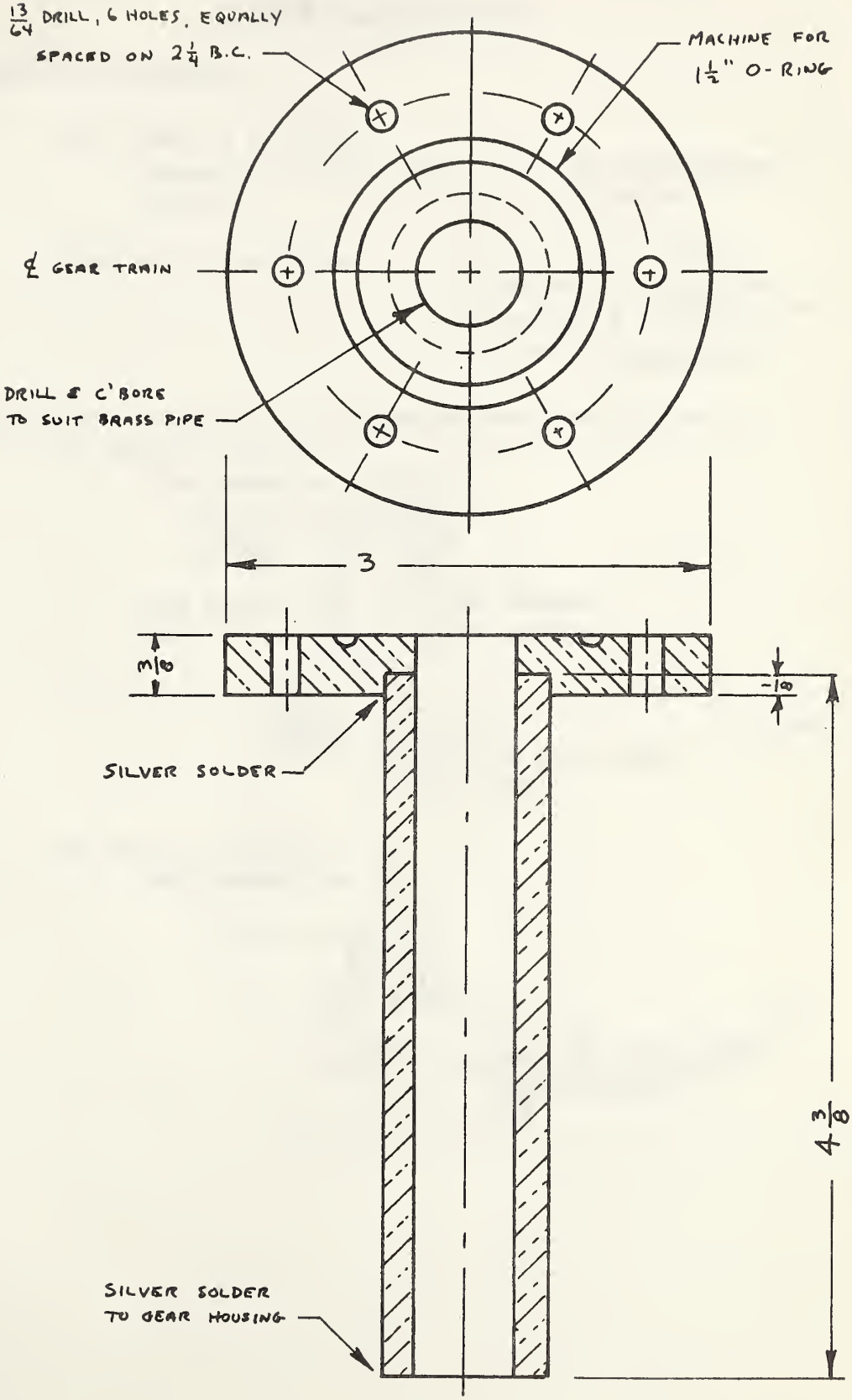


FIGURE I-9 GEAR HOUSING - SUPPORT

Scale: Full Size



APPENDIX II

SAMPLE CALCULATIONS

a) Absolute viscosity

For Fluid A @ 77°F

kinematic viscosity = 122.31 centistokes
density = 0.8732 gm/cc

Absolute viscosity = 122.31 x 0.8732
= 106.80 centipoise
= $106.80 \times 6.72 \times 10^{-4}$
= 0.0717 lb_m/sec ft

b) Instrument Constant (calculated for Fluid B-Run 3)

1) Shear rate:

from equation (28)

$$-\left(\frac{dV}{dR}\right)_{R_b} = \frac{4\pi S R_c^2}{R_c^2 - R_b^2}$$

and since R_b = 0.760 inches
and R_c = 0.850 inches

$$-\left(\frac{dV}{dR}\right)_{R_b} = (62.25)\ S \qquad \text{(II-a)}$$
$$= 62.25 \times 1.46 \text{ RPS}$$
$$= 91.0 \text{ sec}^{-1}$$

2) Shear stress

from equation (1)

$$\tau = -\frac{\mu}{g_c}\frac{dV}{dX}$$
$$= -\frac{\mu}{g_c}\left(\frac{dV}{dR}\right)_{R_b} \qquad \text{(II-b)}$$
$$= \frac{-0.0546\ lb_m/\text{sec ft} \times -91.0/\text{sec}}{32.17\ lb_m ft/lb_f sec^2}$$
$$= 0.1545\ lb_f/ft^2$$

3) Instrument Constant
from equation (27)

$$\tau = a\theta \qquad \text{(II-c)}$$

$$\therefore a = \frac{\tau}{\theta}$$

$$= \frac{0.1545}{18.65}$$

$$= 0.00828 \ lb_f/ft^2 \text{ degree}$$

c) Instrument constant determined graphically

combining equations (II-a) and (II-b) gives

$$\tau = \frac{\mu}{g_c} 62.25 S \qquad \text{(II-d)}$$

combining (II-d) and (II-c) gives

$$a\theta = \frac{\mu}{g_c} 62.25 \ S$$

or $$a = \frac{\mu \ 62.25}{g_c} \frac{S}{\theta}$$

$$= \frac{\mu(62.25)}{g_c \ q} \qquad \text{(II-e)}$$

where q = the slope of the curve relating angular deflection and rotational speed for the appropriate fluid.

for Fluid B from Figure 28

q = 12.7 degrees / RPS

also for Fluid B $\frac{\mu}{g_c} = \frac{0.0546}{32.17} = 0.0017$

therefore from II-e

$$a = \frac{0.0017 \ (62.25)}{12.7} = 0.00833 \ lb_f/ft^2 \text{degree}$$

APPENDIX III

DETERMINATION OF INSTRUMENT CONSTANT FROM PREVIOUS WORK

Ritter (R-46) reported an instrument constant of a = 13.7×10^{-4} lb_f/ft^2 degree, for which the torsion wire length was 10 inches, the bob immersion depth was 4.225 inches, and the bob diameter was 1.582 inches. A 24 B & S gauge MacMichael Viscometer Torsion Wire was used.

Ritter also showed that the instrument constant is given by:

$$a = \frac{\pi r^4 G}{720 L R^2 d} \qquad (a)$$

where a = instrument constant

G = shear modulus of the wire

r = radius of the torsion wire

R = radius of the bob

L = length of the torsion wire

d = depth of immersion of the bob.

If the bob is susported by two torsion wires, then, by a moment balance on the system it can be shown that the applied torque is opposed by a restoring torque in each wire. Thus:

$$\tau RA = 2\gamma_r \qquad (b)$$

where τ = the shear stress on the bob wall

R = the radius of the bob

A = the surface area of the bob

and γ_r = the restoring torque in a single wire.

Ritter has shown that

$$\gamma_r = \frac{\pi^2 r^4 G D}{360 L} \qquad (c)$$

and that $A = 2 \pi R d$ (d)

Substitution of equations (c) and (d) into (b) gives after rearrangement:

$$\tau = \frac{\pi r^4 G D}{360 L R^2 d} \qquad (e)$$

Hence, by comparing equation (e) and equation (27) an instrument constant, a, for the bob supported by two wires is given by:

$$a = \frac{\pi r^4 G}{360 L R^2 d} \qquad (f)$$

For the prototype viscometer, in which the bob is suspended by two wires, the length of each wire, L, is 5.0 inches, and the bob radius R is 0.760 inches. The bob immersion depth may be taken as the length of the cylindrical section of the bob, plus the equivalent length of the conical ends. The equivalent length of one end is not known, but may be estimated from Ritter's work (R-46). Since the dimensions of the bob used in the present work and the bob used in the Alberta Viscometer are very similar, it is assumed that the two bobs will have the same end-effects. For the Alberta Viscometer, Ritter experimentally determined the equivalent

length of the conical section as 0.225 inches. In this study, the bob has two conical ends, therefore the equivalent length of the two ends is taken as 0.450 inches. Since the height of the cylindrical section is 2.625 inches, the bob immersion depth, d, is taken as 3.075 inches.

The term $\pi r^4 G$ in equation (f) is a function of the torsion wire properties only, and can be evaluated from equation (a) using Ritter's data. The value for a 24 B & S gauge wire was found to be

$$\pi r^4 G = 104.3 \text{ lb}_f \text{in}^4/\text{ft}^2 \text{degree}$$

Substituting the appropriate values in equation (f) then yields

$$a = \frac{104.3}{360\,(5)\,(1.520)^2\,(3.075)}$$

$$= 81.6 \times 10^{-4} \text{ lb}_f/\text{ft}^2 \text{degree}$$

Thus for the prototype viscometer the instrument constant should be 81.6×10^{-4} lb_f/ft^2 degree.

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